











# **Analytical Reagents—Standards and Tests**

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## **Standards and Tests**

COMPILED BY  
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From Books of Reference, the various National Pharmacopœias, Papers appearing in Chemical Journals and controlled by the experience gained in the Works and Laboratories of Hopkin & Williams, Limited

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## Preliminary Note

The tests described in the following pages are not intended to replace entirely the usual qualitative and quantitative tests necessary for the identification and determination of the substances under which they are described. They are rather intended to indicate the degree of purity which experience has shown to be necessary, and which may be expected in commerce for chemical reagents used in analysis. For the most part they are limit-reactions—for example, under the conditions described as to concentration, time, temperature, etc., no precipitate or other visible changes take place in the reaction-mixture.

It is hardly necessary to insist here upon the misleading trade description "Chemically Pure," etc., since the substances so described can be shown to contain foreign substances if the tests applied be performed with sufficient delicacy upon a sufficiently large quantity of the substance so described. Broadly speaking, however, chemical reagents answering the descriptions and tests given herein will correspond to the quality described as "Chemically Pure."

The main object of the tests is to ensure the absence of impurities in sufficient proportions to introduce appreciable errors in analytical processes in which they may be employed. The tests described are not always, in themselves, sufficient to ensure the absence of unusual impurities: they have reference rather to such impurities as may be expected to occur from our knowledge of the origin or method of manufacture of the material in question.

In using the tests it should be noted that the reagents employed should themselves correspond to the standards of purity described in this book, and when a solution of a substance is described, the solution described in the "List of Reagents" (see p. 89) is always intended to be used. In this "List of Reagents" the concentration of the solutions is not

fixed upon an arbitrary basis of percentage, but some fraction of the molecular weight of the substance in question is contained in a litre of the solution, that is, the solutions are approximately normal solutions. For qualitative reactions it is sufficient to weigh carefully the indicated quantity of substance, and dissolve it in the solvent to the required volume. Equal volume of the various solutions will thus be equivalent or will bear some simple relationship to each other. The advantage of this system is particularly obvious in the case of acid and alkaline solutions which may be used alternately to render a liquid acid or alkaline, since the volume of an acid solution added as a reagent may be afterwards neutralised with certainty by the addition of an equal volume of the alkaline solution and *vice versa*.

The brief notes under "Trade Varieties" are intended to indicate the nature and composition of chemicals met with in trade, and will be amplified in subsequent issues if the information proves acceptable to readers.

The Reagents complying with the tests described may be obtained from Messrs. Hopkin & Williams, Ltd., by adding the word "Reagent" to the name of the chemical, or more briefly the capital letter "R.": thus Sodium Carbonate R. If ordered through a dealer the letters in brackets (H. & W.) should be added. All the substances so described are prepared and purified by Messrs. Hopkin & Williams, Ltd., and are issued with the label, of which a facsimile is appended.





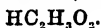


# Analytical Reagents—Standards and Tests

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The abbreviation "Mil," which is recognised by the Board of Trade, is used throughout to indicate a millilitre in preference to the abbreviation "C.c." The latter term is not strictly accurate, since a cubic centimetre is not the thousandth part of a litre of water at 15° C., the temperature at which measure-glasses are graduated. Moreover, the sound of the spoken word "Mil" is more agreeable than the two-syllable sound of "C.c." Specific gravities at 15·5°/15·5° C.

## ACETIC ACID, GLACIAL.



Specific gravity, 1·056; boiling-point, 119° C. Solidifies when cooled, and remains solid below 15·5° C. Contains 99·5 per cent. acetic acid.

### Tests.

*Residue*: 10 mils leave no weighable residue when evaporated in glass dish on a sand-bath.

*Sulphuric Acid*: A 1 in 10 solution should show no turbidity after addition of barium chloride, warming, and setting aside for two hours.

*Sulphurous Acid*: To the above liquid, if it remains clear, or to the filtrate, if a turbidity be produced, add bromine water; no precipitate should appear on standing.

*Hydrochloric Acid*: A 1 in 10 solution should show no cloudiness on addition of silver nitrate solution.

*Metals*: A 1 in 20 solution must show no precipitate or turbidity on addition of an equal volume of hydrogen sulphide solution; or on neutralisation with ammonia and addition of ammonium sulphide solution.

*Empyreumatic Substances*: Foreign organic substances may be detected by their reducing action on permanganate,

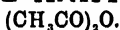
since acetic acid is without action upon this substance. A 1 in 10 solution should be tinted pale pink by N/10  $\text{KMnO}_4$ , and the colour should not be discharged within twenty minutes.

#### Trade Varieties.

Acetic acid is sold in various strengths, but those chiefly met with are "strong" acetic acid (*Acidum Aceticum*, B.P.), 33 per cent., and two varieties of glacial acetic acid known as "50°" and "60°" respectively, the figures indicating the temperatures at which the solidified acid melts. The former contains about, or not less than, 97 per cent. real acid. The specific gravity of the pure acid is increased by the addition of water, until it attains a maximum of 1.0748, corresponding to about 80 per cent. real acid. Further addition of water reduces the specific gravity, and an acid of 43 per cent. has the same specific gravity as the pure acid, 1.056. Only the 60° acid is suitable for the Valenta test.

When an acid short of glacial strength is required the 80 per cent. acid is convenient, as it is not liable to solidify in cold weather.

### ACETIC ANHYDRIDE.



Specific gravity, 1.080; boiling-point, 138°.

#### Tests.

*Residue*: 10 Gm. should leave no residue when evaporated in a glass dish on a sand-bath.

*Hydrochloric Acid*: 2 mls shaken with 50 mls of water should show no turbidity on addition of silver nitrate.

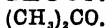
*Distillation Test*: Practically the whole should distil between 137° and 139°.

*Strength*: An article of the above description will contain 95-98 per cent. acetic anhydride, the remainder being acetic acid.

#### Trade Varieties.

The acetic anhydride of technical purity usually shows a slight reaction with silver nitrate, and commences to boil at a lower temperature than that indicated above, owing to the presence of acetic acid. It usually contains about 90 per cent. acetic anhydride and 10 per cent. acetic acid.

### ACETONE.



Specific gravity, 0.797-0.798; boiling-point, 56°-58°. Colourless. Miscible in all proportions with ether, alcohol, and water. Does not dissolve potassium hydroxide or anhydrous calcium chloride.





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**Tests.**

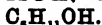
*Residue*: 20 mls should leave no residue on evaporation over a water-bath.

*Reaction*: Should be neutral to litmus.

*Organic Impurities*: Tinted faintly pink by the addition of permanganate, the colour should not be discharged in from five to ten minutes.

**Trade Varieties.**

Acetone is widely used technically as a solvent, and can usually be obtained fairly pure. It has usually been purified by fractional distillation, and the fractions boiling above 60° constitute what is known as "acetone oil." This consists chiefly of higher homologous ketones, and is so called because it is not miscible freely with water like pure acetone. Acetone oil finds a use as a solvent in the varnish industry.

**ALCOHOL, AMYL.**

Specific gravity, 0.814 to 0.815; boiling-point, 128°-132°. Soluble in about 40 parts of water; miscible with benzene or petroleum ether.

**Tests.**

*Residue*: 10 mls leaves no residue after evaporation in a glass dish over a water-bath.

*Furfural*: Not more than a pale yellow or reddish-brown tint should be produced by shaking it with an equal volume of strong sulphuric acid.

**Trade Varieties.**

Fusel oil is commonly known as "commercial" amyl alcohol, but is a most variable mixture of both lower and higher homologues with water. Fusel oil on distillation yields first a watery fraction containing some of the lower homologues in solution.

**ALCOHOL, ETHYL.**

Specific gravity, 0.796; boiling-point, 78.3°. Miscible with water in all proportions. Colourless. Very hygroscopic. When mixed with water a contraction occurs, the maximum contraction being shown by a mixture in the proportion of one molecular weight of alcohol and three molecular weights of water.

**Tests.**

*Residue*: Should leave no residue on evaporation.

*Odour*: Should give no foreign odour when allowed to evaporate from white filter paper, or on dilution with water.

*Aldehyde*: If a few drops of silver nitrate solution be added to 10 mls of the alcohol, and the mixture warmed and set

aside for twenty-four hours, no turbidity or blackening should result. When mixed with half its volume of 5 per cent. potassium hydroxide solution, no darkening should immediately occur.

*Reaction*: Should show no reaction with blue litmus paper.

#### Trade Varieties.

Absolute alcohol can be obtained containing not less than 99.5 per cent. ethyl alcohol. A weaker variety containing 97 per cent. is sometimes sold as "absolute," but is readily distinguished by its specific gravity, which is about 0.80. In the U.S.P., alcohol of 95 per cent. by weight is official. In this country "rectified spirit" is generally understood to be the *Spiritus Rectificatus* of the B.P., which contains 90 per cent. by volume of ethyl alcohol, and has a specific gravity of 0.834.

## ALCOHOL, METHYL.



Specific gravity, 0.796; boiling-point, 65°. Clear, colourless fluid, miscible with water.

#### Tests.

*Residue*: Should leave no residue on evaporation.

*Acetone*: No yellow colour or turbidity or odour of iodoform should be produced by the addition of caustic soda solution, and a few drops of N/10 iodine solution to 5 mls of the alcohol diluted with 40 mls water.

*Colour Test*: Addition of large excess of strong caustic soda solution should produce no coloration, and only the faintest yellow colour should be produced by the gradual addition of an equal volume of strong sulphuric acid.

*Distillation Test*: Practically the whole of the alcohol should distil within 1°.

#### Trade Varieties.

Methyl alcohol, as described above, will contain not more than 0.05 to 0.10 per cent. of acetone, and is suitable for use in making up stains of eosin-methylene blue (Jenner, Leischmann, Romanowsky). Another variety, known as "pure methyl alcohol," contains 1 to 2 per cent. acetone, and has a boiling-point ranging over several degrees in consequence. In commerce wood naphtha is obtainable, containing 70 per cent. upwards of methyl alcohol, the remainder being acetone, methyl acetate, allyl compounds, water, etc. A purified and rectified wood naphtha is known as pyroxylic spirit.

## AMMONIUM CARBONATE.

In white, translucent, flattened cakes. Composition variable, but consisting, when freshly sublimed, of ammonium carbonate and carbamate. When exposed, it loses am-





monia and leaves a white powdery residue of ammonium hydrogen carbonate.

### Tests.

*Residue*: Should dissolve completely in 10 parts of distilled water, and leave no residue when heated in a platinum dish.

*Sulphuric or Hydrochloric Acid*: A 5 per cent. solution in water, after addition of slight excess of pure nitric acid, should show no turbidity on addition of barium chloride or silver nitrate, and standing one hour.

*Iron*: A 5 per cent. solution supersaturated with pure nitric acid should show only the faintest pinky tint on the addition of solution of ammonium sulphocyanide. It is desirable to make a blank test with the diluted nitric acid, as this is liable to contain traces of iron, particularly if stored for any length of time after redistillation.

*Metals*: A 2 per cent. solution faintly acidulated with acetic acid should show no darkening with an equal volume of hydrogen sulphide solution.

*Tarry Substances*: A 5 per cent. solution slightly acidified with sulphuric or nitric acid should have no tarry odour, and the solution evaporated to dryness in a porcelain dish should leave a colourless and odourless residue.

### Trade Varieties.

The ordinary ammonium carbonate of commerce in lumps sometimes leaves a minute residue on ignition. It may contain small traces of sulphate or chloride, and nearly always shows a distinct pink tint when tested for iron as above. Occasionally distinct traces of tarry substances are present, the odour of which is masked by the strong odour of the ammonia and only detectable by the nose after neutralisation with sulphuric acid. The formula usually ascribed to this substance indicates 32.6 per cent.  $\text{NH}_3$ , but this standard is never attained, 31 per cent. being about the average of good commercial specimens when freed from the efflorescent coating of bicarbonate. Volcanic ammonium carbonate was formerly obtained in making borax from the boric acid of volcanic origin from Tuscany, the ammonia being expelled when the acid was treated with sodium carbonate. It was much prized on account of its freedom from tarry impurities, but is not now a commercial product.

## AMMONIUM CHLORIDE.



White granular or powdery crystals free from odour and soluble in 2 parts of water and in about 50 parts of alcohol.

### Tests.

*Residue and Tarry Impurities*: 5 grammes moistened with nitric acid and evaporated on a water-bath to dryness in a

platinum crucible should leave a white saline residue. On heating this with the flame it should volatilise, leaving less than 0.5 Mg. residue, and the substance should remain colourless during volatilisation.

*Sulphate*: A 10 per cent. solution should show no turbidity after addition of 1 mil barium chloride solution and standing two hours.

*Metals*: A 10 per cent. solution should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron*: A 5 per cent. solution should not show any immediate blue colour on the addition of one drop of potassium ferrocyanide solution. Even the best obtainable samples will seldom stand a more stringent test than this.

*Thiocyanates*: The addition of a few drops of ferric chloride solution to a 5 per cent. solution acidulated with hydrochloric acid should not produce an immediate red colour.

*Organic Impurities*: A 5 per cent. solution rendered pink by the addition of N/10 potassium permanganate solution should be only slowly decolorised and should not become brown.

#### Trade Varieties.

This salt is met with in many forms. Sal ammoniac is the name applied to the sublimed substance in crystalline lumps, showing a columnar stratification on fracture. When crushed to small pieces it is used for charging Leclanché batteries. A commercial form of granular crystals is also sold, as well as another form of crushed crystals made to resemble crushed sal ammoniac, and known as dog-tooth crystals. These varieties usually contain more or less of the impurities mentioned above and always considerable traces of iron and fixed impurities. The less pure samples have a reddish or greyish tint.

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## AMMONIUM FLUORIDE.



Colourless translucent crystals, very soluble in water. The aqueous solution has an acid reaction owing to the easy dissociability of the salt. It attacks glass, and should be stored in vessels of gutta percha or paraffin wax. Glass bottles may be used if first thickly coated internally with paraffin wax and closed with a stopper of the same material.

#### Tests.

*Residue*: 5 grammes ignited in a platinum dish should leave not more than 0.2 Mg. residue.

*Chlorides*: The addition of silver nitrate and nitric acid to a 5 per cent. solution should give no turbidity.

*Sulphate and Silicofluoride*: A 5 per cent. solution







strongly acidulated with hydrochloric acid should give no turbidity on the addition of barium chloride solution.

*Metals*: The addition of ammonia and ammonium sulphide or an equal volume of hydrogen sulphide solution to a 5 per cent. solution of the salt should not produce a black coloration or turbidity.

#### Trade Varieties.

The article described above can only be produced by neutralising very pure hydrofluoric acid with pure ammonium hydroxide or carbonate in a platinum dish. Commercial ammonium fluoride made from crude acid is often colourless, but contains considerable quantities of sulphate, fluosilicate, and lead, as well as arsenic and other impurities.

## AMMONIUM HYDROXIDE.

- A solution of ammonia gas in water. In this country Liquor Ammoniae Fortis sp. grav. 0.880 is usually sold. This corresponds to a strength of about 35 per cent.  $\text{NH}_3$ , but owing to the ease with which ammonia escapes from such strong solutions the product will usually be found slightly under this strength.

#### Tests.

*Residue*: 20 mls evaporated on a water-bath in a glass dish should leave a residue weighing not more than 0.5 Mg.

*Pyridine and Tarry Impurities*: 10 mls of the liquid is diluted with 40 mls of water, a few drops of methyl orange solution added, and then sufficient pure nitric acid to nearly neutralise the ammonia. The liquid should not change colour, and should be free from unpleasant odour. On evaporation on a water-bath it should leave a white residue.

*Chlorides and Sulphates*: 5 mls diluted with 20 mls of water and rendered slightly acid with pure nitric acid should show only the faintest turbidity on adding silver nitrate or barium chloride solution, warming, and setting aside for three hours.

*Carbonate*: 5 mls should form a clear mixture with 20 mls of lime water, and even after boiling only the faintest turbidity should be produced.

*Metals*: 5 mls diluted with 25 mls of water should show no discolouration on the addition of 5 drops of ammonium sulphide solution.

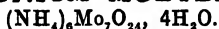
*Other Impurities*: The absence of residue exceeding the amount given above will exclude appreciable traces of other probable impurities.

#### Trade Varieties.

The ordinary Liq. Ammon. Fort. of commerce is of variable quality. It sometimes shows considerable traces of tarry impurities and traces of iron. The solid residue, when exceeding in amount that mentioned above, often con

tains considerable proportions of magnesia. The British Pharmacopœia strong solution is described as of sp. gr. 0·891 and containing 32·5 per cent.  $\text{NH}_3$ . A solution of this sp. gr. will, however, contain only 31·5 per cent. In the U.S.P. a strong solution containing 28 per cent.  $\text{NH}_3$  is official, and in the Ph. Ger. one containing 20 per cent.  $\text{NH}_3$ . A very crude product is sold for ice-making.

## AMMONIUM MOLYBDATE.



Colourless dense crystalline masses which evolve ammonia on careful ignition and leave a white residue of about 80 per cent. molybdic anhydride. It should dissolve without residue in 10 parts of water.

### Tests.

*Phosphate*: This test is most important since the reagent is used for the detection and determination of phosphoric acid. Dissolve 5 grammes in 20 mils of water and 10 mils strong ammonia solution. Add this liquid very gradually, with constant stirring, to a mixture of 75 mils each strong nitric acid and water. Warm the product and set aside for one to two hours. No yellow precipitate should be produced.

*Metals*: 1 gramme dissolved in 10 mils of water and rendered distinctly alkaline with ammonia should show no darkening in colour after the addition of 5 drops of ammonium sulphide solution.

*Sulphates and Chlorides*: A 10 per cent. solution acidulated with nitric acid should show no turbidity after the addition of  $\frac{1}{2}$  its volume of solution of barium chloride or silver nitrate and setting aside two hours.

### Trade Varieties.

The ordinary ammonium molybdate of commerce is usually sufficiently pure for analytical purposes if it has a well-crystallised appearance and has only a slight shade of green.

## AMMONIUM NITRATE.



Colourless crystals, readily fusing to a colourless fluid when heated, and freely soluble in water.

### Tests.

*Residue and Tarry Impurities*: 5 grammes heated cautiously with 5 drops of nitric acid in a platinum crucible should melt to a colourless fluid, and on continued heating to a low red heat should leave a residue weighing less than 0·5 Mg.

*Sulphates or Chlorides*: 10 mils of a 10 per cent. solution faintly acidulated with nitric acid should show only a faint turbidity after addition of 1 mil of solution of barium chloride or 1 mil silver nitrate and standing two hours.





**Metals:** A 10 per cent. solution should show no darkening in colour after addition of an equal volume of hydrogen sulphide solution.

**Iron:** A 5 per cent. solution should not show any immediate blue colour on the addition of 1 drop of potassium ferrocyanide solution.

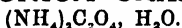
**Thiocyanates:** The addition of a few drops of ferric chloride solution to a 5 per cent. solution of the salt acidulated with hydrochloric acid should not produce an immediate red colour.

**Nitrites:** 50 mls of a 2 per cent. solution acidulated with 1 mil dilute sulphuric acid should show no yellow-brown coloration after adding 1 mil of a  $\frac{1}{2}$  per cent. freshly prepared solution of metaphenylenediamine hydrochloride and standing ten minutes.

#### Trade Varieties.

Commercial ammonium nitrate in white, or nearly white, crystalline masses usually contains sulphates and chlorides, and leaves a considerable residue on ignition.

### AMMONIUM OXALATE.



Colourless needle crystals, soluble in water, forming a neutral solution.

#### Tests.

**Solubility:** 1 gramme should dissolve without residue in 40 mls of water.

**Residue:** 3 grammes heated to a low redness in a platinum dish should leave not more than 0.5 Mg. residue.

**Sulphates:** 50 mls of a 2 per cent. solution with 2 mls hydrochloric acid and 2 mls barium chloride solution should show no turbidity after warming and setting aside for four hours.

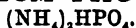
**Chlorides:** 50 mls of a 2 per cent. solution with 1 mil nitric acid and 2 mls silver nitrate solution should show no turbidity after standing two hours.

**Heavy Metals and Iron:** 50 mls of a 2 per cent. solution with 1 mil solution of ammonia and 2 mls ammonium sulphide solution should show no darkening after standing one hour.

#### Trade Varieties.

These are usually of good quality, but often incompletely soluble in water, show distinct traces of sulphates and iron, besides leaving some residue after ignition.

### AMMONIUM PHOSPHATE.



Colourless crystals, entirely soluble in 4 part of water, forming a faintly alkaline solution; insoluble in alcohol.

#### Tests.

**Sulphates and Chlorides:** 10 mls of a 5 per cent. solution acidulated with 5 decimils of nitric acid should show no tur-

bidity after addition of 1 mil barium chloride or silver nitrate solution and standing one hour.

*Fixed Alkalies:* Dissolve 2 grammes in 50 mils of water, warm, and add drop by drop a solution of lead acetate until the latter is in slight excess. Allow to stand twenty minutes, filter, and add 1 drop of lead acetate solution to the filtrate, which should show no turbidity. Precipitate the lead from the filtrate with hydrogen sulphide, warm, and filter again. The filtrate should leave less than 0.5 Mg. of residue on evaporation and incineration.

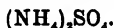
*Arsenic:* 2 grammes after thirty minutes' treatment by the Marsh-Berzelius test should yield no mirror of arsenic.

*Metals:* 20 mils of a 5 per cent. solution with 1 mil ammonium sulphide solution should show no darkening after standing thirty minutes.

#### Trade Varieties.

These are met with in very varying degrees of purity. Arsenic is often present in considerable traces, and they usually contain sulphates and traces of iron. Ammonium phosphate, like sodium phosphate, although acid in constitution, forms an alkaline solution in water. It should be carefully distinguished from the biphosphate  $\text{NH}_4\text{H}_2\text{PO}_4$ .

## AMMONIUM SULPHATE.



Colourless crystals, soluble in water, but (like other inorganic sulphates) insoluble in alcohol.

#### Tests.

*Residue:* 5 grammes should leave on incineration a residue weighing less than 0.5 Mg.

*Chlorides:* A 10 per cent. solution acidulated with nitric acid should show no turbidity within thirty minutes after adding 1/10 its volume of silver nitrate solution.

*Metals:* A 10 per cent. solution should give no precipitate or darkening in colour after addition of 1/10 its volume of ammonium sulphide solution.

*Thiocyanates:* A 10 per cent. solution with 1/20 its volume of hydrochloric acid should not give a red coloration after addition of a few drops of ferric chloride solution.

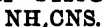
#### Trade Varieties.

A crude variety, for agricultural purposes, is met with in varying degree of purity. It is obtained as a by-product in purifying gas-liquors, and the colour varies from grey to white. This contains more or less of all the impurities mentioned above.







**AMMONIUM THIOCYANATE.**

Colourless, transparent, easily fusible crystals, very soluble in alcohol and water. The salt is extremely sensitive to contamination with iron derived from the vessels in which it is stored or from dust falling upon it through careless storage.

**Tests.**

*Solubility:* The salt should be entirely soluble in 10 parts of water or absolute alcohol.

*Residue:* 2.5 grammes should leave on incineration a residue weighing less than 0.5 Mg.

*Sulphates:* A 10 per cent. solution should give no turbidity after addition of barium chloride solution and standing twenty minutes.

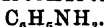
*Metals:* A 10 per cent. solution should show no darkening after adding a few drops of ammonium sulphide solution.

*Iron:* The presence of this impurity is usually indicated by a pinkish tint in the crystals. Even if colourless a 10 per cent. solution should remain colourless after addition of 1/10 its volume of nitric acid.

**Trade Varieties.**

The commercial salt usually shows traces of sulphates, and is often pink from traces of iron. A yellow tint is indicative of tarry impurities, while lead is occasionally present in colourless samples.

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**ANILINE.**

Nearly colourless fluid. Sp. gr. 1.027. Completely soluble in 35 to 40 parts of water; more soluble in alcohol, ether, and fixed and volatile oils.

**Tests.**

*Boiling Point:* Pure aniline should completely distil between 183° and 184°.

*Other Tests:* The sp. gr. and complete solubility in water and dilute hydrochloric acid to a colourless fluid are sufficiently indicative of pure aniline.

**Trade Varieties.**

Commercial aniline, or "aniline oil," usually has a wide range of boiling point. Unless freshly distilled it is yellowish-brown in colour, but is still sufficiently good for use in preparing microscopical staining solutions.

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**ARSENIOUS ANHYDRIDE.**

In glassy, translucent lumps, which become opaque and porcelain-like on keeping; or in fine white powder, obtained by grinding. The glassy variety is more soluble than the opaque modification, but both should dissolve by long treatment

in 15 parts of boiling water. It is much more readily soluble in presence of acid and alkalies.

#### Tests.

*Residue*: 2 grammes should leave on ignition a residue weighing less than 0.5 Mg.

*Earthy Impurities*: 1 gramme should dissolve completely in 20 mls of 10 per cent. ammonia solution.

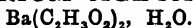
*Antimony, Tin, or Cadmium*.—0.5 gramme dissolved in 50 mls of water with 1 mil hydrochloric acid gives a yellow precipitate with hydrogen sulphide, which, after collection and once washing with distilled water, should be completely soluble in ammonium carbonate solution.

*Quantitative Test*: Dissolved in sodium bicarbonate solution and titrated with N/10 iodine solution, it should show 0.98 to 100 per cent.  $\text{As}_2\text{O}_3$ .

#### Trade Varieties.

White arsenic is met with in commerce titrating 98-99 per cent.  $\text{As}_2\text{O}_3$ . It leaves a little residue on ignition, and dissolves in strong caustic soda solution, usually forming a brownish solution. Lower grades of crude arsenic having a grey tint, containing about 95 per cent.  $\text{As}_2\text{O}_3$  and contaminated with metallic and fixed earthy impurities, and occasionally with arsenic and sulphuric acids, are also met with.

### BARIUM ACETATE.



Small white crystals, soluble in 3 parts of water.

#### Tests.

*Sulphates*: 1 gramme should dissolve completely and rapidly in 20 mls of water, forming a bright solution.

*Chlorides*: A 5 per cent. solution should show no turbidity within thirty minutes after acidulation with nitric acid and addition of silver nitrate solution.

*Alkalies*: 1 gramme is dissolved in 50 mls water with 0.5 mil hydrochloric acid, and the barium precipitated by the addition of 10 mls dilute sulphuric acid (1 in 10). After warming, set aside several hours, filter, evaporate the filtrate to dryness in a platinum dish, and ignite. If there be any residue its weight should not exceed 1.0 Mg.

#### Trade Varieties.

These usually contain more alkalies than is allowed by the test above, and are not soluble in water to an absolutely clear solution owing to the presence of traces of sulphate.

### BARIUM CARBONATE.



White powder, insoluble in water, but soluble with effervescence in dilute hydrochloric and nitric acids. In the case of





nitric acid, the nitrate of barium produced is only sparingly soluble in presence of excess of nitric acid.

#### Tests.

*Sulphates*: 2 grammes suspended in 50 mils of water should be dissolved to a perfectly clear solution by a slight excess of hydrochloric acid.

*Chlorides*: 1 gramme suspended in 20 mils of water is dissolved by a slight excess of nitric acid. After addition of 1 mil silver nitrate solution and setting aside, no turbidity should appear within two hours.

*Nitrates*: 1 gramme is suspended in 10 mils of water and dissolved by addition of slight excess of acetic acid. The liquid is tinted blue by the addition of indigo solution, and 5 mils strong sulphuric acid added. The colour should not rapidly fade.

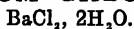
*Alkalies*: 1 gramme is suspended in 50 mils of water, and dissolved by a slight excess of hydrochloric acid. This solution should comply with the test for alkalies as described under Barium Acetate.

*Ammonium Salts*: 1 gramme shaken in a test-tube with 5 mils of water, solution of potash or soda added, and the mixture gently heated. No ammonia vapours should be evolved.

#### Trade Varieties.

Native barium carbonate is used in considerable quantities for industrial purposes, and usually contains considerable proportions of earthy impurities, iron, etc. For laboratory use two varieties are commonly met with—(1) "precipitated by soda," (2) "precipitated by ammonia." These are prepared by precipitating a solution of barium nitrate or chloride with carbonate of sodium or ammonium respectively, and always retain alkaline salt, however prolonged may be the washing to which they have been subjected. The second variety is employed as a reagent, except when absolute freedom from ammonia is required.

## BARIUM CHLORIDE.



Colourless, transparent crystals, very soluble in water.

#### Tests.

*Sulphates*: 2 grammes should dissolve in 10 mils of water, forming a bright solution, free from any opalescence.

*Alkalies*.—See under Barium Acetate.

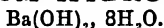
*Nitrates*: 1 gramme is dissolved in 10 mils of water, and the solution tinted blue by indigo solution. The colour should not rapidly fade after addition of 5 mils strong sulphuric acid.

*Heavy Metals*: A 5 per cent. solution should show no darkening in colour or precipitate on the addition of hydrogen sulphide or ammonium sulphide solution.

**Trade Varieties.**

Commercial barium chloride is usually of good quality, although a great variety of impurities have at times been noticed in it. It usually contains iron and alkalies. The recrystallised salt, unless prepared with the greatest care, may contain traces of sulphate, shown by the faint opalescence of its solution in water.

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**BARIUM HYDROXIDE.**

White crystals, becoming opaque on keeping, soluble in 20 parts of water, forming a strongly alkaline solution, which may be slightly opalescent from the presence of traces of carbonate.

**Tests.**

*Sulphates:* 1 gramme should form a bright solution, free from opalescence in 30 mils of water after addition of a slight excess of nitric acid.

*Chlorides:* The clear solution formed in testing for sulphates should show no opalescence after adding 2 mils silver nitrate solution and setting aside for thirty minutes.

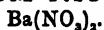
*Sulphides:* 1 gramme is dissolved in 20 mils of water, with the addition of a distinct excess of hydrochloric acid. The solution on warming should not smell of hydrogen sulphide or be darkened by the addition of lead acetate solution.

*Lead and Other Metals:* 1 gramme dissolved in 30 mils of water, with a slight excess of hydrochloric acid, should show no darkening on addition of an equal volume of hydrogen sulphide solution.

**Trade Varieties.**

Commercial "barium hydrate" contains some carbonate and more or less sulphate, sulphite, sulphide, and chloride. A 5 per cent. solution is used under the name "Baryta water" as a dressing for the preservation and hardening of decayed stonework.

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**BARIUM NITRATE.**

Colourless transparent crystals soluble in about 20 parts of water, much less soluble in presence of acids.

**Tests.**

*Sulphates:* 1 gramme should dissolve in 30 mils of water, forming a perfectly clear solution free from any trace of opalescence.

*Chlorides:* The clear solution formed in testing from sulphates, after addition of 2 drops of nitric acid and 2 mils







silver nitrate solution, should remain bright after setting aside two hours.

*Alkalies and Heavy Metals:* These are tested for as described under Barium Acetate.

#### Trade Varieties.

Commercial barium nitrate is usually of good quality, but may contain iron and sulphates, and sometimes lead.

## BENZENE.



Colourless, inflammable, mobile fluid. Insoluble in water, easily soluble in absolute alcohol and ether. Boiling-point  $80.5^\circ$ . Solidifies when cooled below  $0^\circ$  to a crystalline mass, which should not again melt until its temperature is allowed to rise to  $4^\circ$ - $5^\circ$ .

#### Tests.

*Residue:* 10 mls evaporated at a low temperature from a glass dish should leave no visible residue.

*Distillation Test:* When 100 mls are submitted to fractional distillation, only a few drops should come over before the thermometer reaches  $80^\circ$ . It should then remain steady between  $80^\circ$  and  $81^\circ$  until all but the last few drops have distilled over.

*Thiophene:* 10 mls shaken with 2.5 mls strong sulphuric acid and 2 drops fuming nitric acid (sp. gr. 1.5) : no green or blue colour should appear in the mixture.

#### Trade Varieties.

The "crystallisable benzene" of commerce complies with the physical properties described above, but is not always free from thiophene. The melting-point of the frozen benzene is a good indication of freedom from more than traces of other hydrocarbons.

The name Benzene should be applied to the pure, or nearly pure, hydrocarbon,  $\text{C}_6\text{H}_6$ . Mixtures of benzene with other hydrocarbons, chiefly the higher homologues of the benzene series, are known in the trade as benzol or benzole. They are sold as "30 per cent.," "60 per cent.," "90 per cent.," etc., the figures indicating not the percentage of pure benzene, but the proportion which distils over below  $100^\circ$ . Such products contain varying proportions of other hydrocarbons, chiefly toluene and xylene. "Rectified benzole" is usually understood to be a redistilled 90 per cent. benzol, and as such should comply with the characters and tests of Benzol B.P. Benzol or benzene should be carefully distinguished from benzin or benzine (*vide* Petroleum Ether), which consists chiefly of hydrocarbons of the aliphatic or paraffin series.

## BISMUTH SUBNITRATE.

A heavy white microcrystalline powder, odourless and tasteless, insoluble in water, soluble in hydrochloric or nitric acid. Moistened with water, it shows a slightly acid reaction with blue litmus paper.

### Tests.

For the first four tests proceed as follows :—Dissolve 3 grammes of the salt in 4 mils of nitric acid, and pour the solution into 100 mils of water. Filter to remove the white precipitate of bismuth oxynitrate, concentrate the filtrate to 30 mils, filter again, and test separate portions of this filtrate as under :—

*Lead* : 5 mils of filtrate with 5 mils of dilute sulphuric acid should not become cloudy on standing fifteen minutes.

*Copper* : If solution of ammonia be added to 5 mils until the fluid has a distinctly alkaline reaction and the precipitate be allowed to settle, the supernatant fluid should show no blue colour.

*Silver* : Add dilute hydrochloric acid to 5 mils of the filtrate. Any precipitate produced by the first few drops should be completely soluble in excess of the reagent.

*Sulphates* : 5 mils of filtrate should give no precipitate with 1 mil barium nitrate solution.

*Residue* : 1 gramme heated to redness until red fumes cease to be evolved leaves a residue of bismuth oxide weighing 0.79 to 0.82 gramme.

*Arsenic* : Ignite 1 gramme and dissolve the residue in warm sulphuric acid. This solution, when examined by the Marsh-Berzelius test, should give no arsenical mirror.

*Selenium or Tellurium* : Dissolve 1 gramme in 1.5 mils nitric acid, add 10 mils ammonium chloride solution, and dilute with water to 100 mils. Filter and to the filtrate add 10 grammes sodium sulphite, and set aside for twelve hours. No red or black colouration or precipitate should be produced.

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## BROMINE.

Heavy dark-brown, mobile fluid, evolving reddish fumes, having a disagreeable and irritating odour (sp. gr. 2.99). Boiling-point, 63°. Soluble in 30 parts of water; freely soluble in alcohol, ether, chloroform, and carbon bisulphide.

### Tests.

*Residue* : 1 mil allowed to evaporate from a porcelain dish, warming towards the end on a water-bath, should leave less than 0.2 Mg. of residue.

*Iodine* : 1 mil is shaken with 50 mils of water in a stoppered flask, and reduced iron or iron powder added in small portions





until the liquid is almost colourless. The filtered fluid, on the addition of 5 drops ferric chloride solution and some mucilage of starch, should not assume a blue colour.

*Organic Compounds:* 1 mil of bromine is shaken in a stoppered flask with 50 mils of water, and sufficient ammonia solution added, slowly and in portions, until the brown colour disappears. No oily drops should separate, and the liquid should be clear and colourless, and leave a white residue of ammonium bromide when carefully evaporated to dryness.

*Sulphates:* 1 gramme of the ammonium bromide, formed in the foregoing test, dissolved in 20 mils water with the addition of 0.5 mil hydrochloric acid, should give no precipitate after the addition of 2 mils barium chloride solution and setting aside three hours.

*Chloride:* 0.1 gramme of the ammonium bromide, formed as described above, is dissolved in 10 mils of water and 4 mils ammonium carbonate solution added. 12 mils N/10  $\text{AgNO}_3$  solution is then added, and the whole well shaken. The filtrate, after being rendered faintly but permanently acid with nitric acid, should show only the faintest opalescence due to the trace of silver bromide dissolved by the ammonium carbonate solution, silver chloride being readily soluble in this reagent.

#### Trade Varieties.

Bromine derived from the Stassfurt deposits is usually of excellent quality, but supplies of American origin are of variable quality, and require careful examination.

## CALCIUM CARBONATE.



White powder, insoluble in pure water but slightly soluble in presence of carbon dioxide.

#### Tests.

*Alkalies:* 1 gramme shaken with 20 mils water and set aside thirty minutes should yield a filtrate neutral to litmus. 10 mils of this evaporated to dryness and gently ignited should leave a residue weighing less than 1 Mg.

*Chlorides:* 1 gramme suspended in 20 mils of water should dissolve, with effervescence, to a clear solution on the addition of a slight excess of nitric acid, and the addition of 2 mils silver nitrate solution should produce no immediate turbidity.

*Sulphates:* 1 gramme suspended in 20 mils of water should dissolve with effervescence to a clear solution on the addition of a slight excess of hydrochloric acid, and the addition of 2 mils barium chloride should give no turbidity, even after warming and setting aside for three hours.

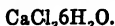
**Magnesia:** 1 gramme is suspended in 20 mls of water and dissolved by the addition of a slight excess of hydrochloric acid. Ammonia is added until the liquid, after well shaking, smells strongly of ammonia, and then 10 mls of ammonium oxalate solution. Warm and set aside two hours; filter, and test the filtrate with a few drops of ammonium oxalate solution to ensure complete precipitation of all calcium as oxalate; the clear liquid should show no turbidity after adding 2 mls sodium phosphate solution and setting aside several hours.

**Metals:** A solution of 1 gramme in 20 mls of water with slight excess of hydrochloric acid should show no discoloration or turbidity (a) on adding twice its volume of freshly prepared hydrogen sulphide solution or (b) on adding a slight excess of ammonia and 5 drops of ammonium sulphide solution.

#### Trade Varieties.

Calcium carbonate occurs in trade in a great variety of forms. Prepared chalk is native calcium carbonate, ground and elutriated to remove gritty particles of silica and other foreign matter. It contains iron as well as the other impurities mentioned above. "Precipitated" chalk is usually prepared from calcium chloride and sodium carbonate, and usually contains more alkali and chloride than is allowed by the tests above. A by-product obtained in water-softening processes and of varying purity also comes on the market, and is also sold as "precipitated" chalk. The purer forms are prepared by precipitation with ammonium carbonate, since the soluble ammonium salt can be more completely washed away from the precipitated calcium carbonate than the corresponding sodium salt. Any trace of ammonium salt left in the precipitate does not interfere, on account of its volatility, with analytical results as much as the sodium salt.

## CALCIUM CHLORIDE, CRYSTALLISED.



White, deliquescent crystals, freely soluble in water and alcohol.

#### Tests.

**Solution in Water:** This should be perfectly clear and neutral in reaction.

**Barium and Strontium:** 1 Gm. dissolved in 10 mls of water should show no turbidity after adding 20 mls of calcium sulphate solution and setting aside six hours.

**Iron and Heavy Metals:** 1 Gm. dissolved in 20 mls of water and 5 drops of ammonia solution added should show no darkening or discoloration on the addition of 5 drops of ammonium sulphide solution.







## **CALCIUM CHLORIDE, FUSED.**

White, translucent crystalline masses obtained by fusing the crystals, so that the water is reduced to about 20 per cent. and casting the product in cakes, which are broken up when cold. It should be completely soluble in water and conform to the same tests as the crystallised form.

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## **CALCIUM CHLORIDE, ANHYDROUS.**

White, porous, deliquescent fragments produced by fusing and heating the crystallised form until most of the water of crystallisation is driven off. The product is then granulated by stirring during cooling, and the desiccation completed in the air-bath. During the drying a little basic salt is usually produced; hence the solution in water may be alkaline. When used for the absorption of moisture in elementary analysis the calcium chloride tube should be first treated with washed and dried carbon dioxide in order to convert any oxide into carbonate, the excess of carbon dioxide being afterwards expelled by a current of dry air.

### **Trade Varieties.**

Crude calcium chloride is met with in commerce in large translucent brownish blocks, packed in sealed iron drums. This variety is used for preparing the brine used in ice machine, where the purity of the salt is of minor importance. A crude form of dried calcium chloride, besides containing the impurities specified above, is usually discoloured, and only partially soluble in water, forming a very alkaline fluid, due to the presence of oxysalt, produced in drying rapidly at high temperature.

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## **CALCIUM OXIDE, QUICKLIME.**



The most suitable form for use as a reagent is obtained by heating clean selected pieces of marble. Quicklime from this source occurs in white lumps, easily broken, and showing a fracture with a granular surface. If exposed to air or stored in vessels insecurely closed the lumps become pulverulent, owing to the absorption of moisture, which results in the formation of the hydroxide, slaked lime. When moistened with water the lumps swell up, crack, and fall to powder with evolution of heat. The rapidity with which this reaction occurs depends upon the density or compactness of the lumps, which, in turn, depend on the variety of marble from which the lime has been made. Black marble yields a very dense and hard lime.

**Tests.**

*Carbonate:* 2 Gm. placed in a porcelain dish and slaked by the addition of water, added a few drops at a time, should fall to a white smooth powder. The slaked lime, suspended in 30 mls of water, should dissolve on the addition of hydrochloric acid without effervescence, yielding at most a few bubbles of gas.

*Silicate:* The liquid obtained in testing for carbonate, and containing a slight excess of hydrochloric acid, should be almost clear, even after boiling.

*Alumina:* 10 mls of the liquid obtained by dissolving the slaked lime in hydrochloric acid, as described above, filtered if necessary, should yield no precipitate on the addition of a slight excess of solution of ammonia.

*Sulphate:* Another 10 mls of the filtrate should give no precipitate on the addition of 1 mil of barium chloride solution, and even after standing one hour not more than the faintest opalescence should appear.

*Chloride:* 1 Gm. of lime, carefully slaked, and the product suspended in 10 mls of water, should dissolve almost entirely on the addition of a slight excess of nitric acid. The solution, filtered if necessary, should remain clear on the addition of 1 mil of silver nitrate solution, and show only the faintest turbidity after standing one hour.

**Trade Varieties.**

- Quicklime from marble is usually of good quality. The surface of the lumps should be clean and show only traces of the fuel or hearths of the furnace on which it has been roasted. Lime from chalk or limestone is less compact, more friable, and its colour varies from nearly white to buff or grey, while the impurities described above are present in variable quantities. It is also usually incompletely soluble in acids and contains iron.

**CALCIUM HYDROXIDE, SLAKED LIME.**

For reagent use this should be obtained by slaking the quicklime from marble. The slaked lime should be finely sifted to remove lumps and thoroughly dried, the product being a smooth white powder. When dry, the slaked lime absorbs carbon dioxide comparatively slowly from the air. Calcium hydroxide is sparingly soluble in water, about 1 in 900, and a saturated solution is known as "lime water." Its solubility is much increased by the presence of sugar.

**Tests.**

These are carried out as described under Calcium Oxide,





except that the powder is suspended at once in water and dissolved by the appropriate acid.

#### Trade Varieties.

See under Calcium Oxide. Sulphates and chlorides may be partly removed by shaking an impure slaked lime with ten times its weight of water and filtering.

## CHARCOAL, PUREST ANIMAL CHARCOAL.

A dry, black, and fine powder.

#### Tests.

*Ash*: 1 Gm. carefully incinerated until a nearly white ash is obtained should leave a residue weighing between 0.05 and 0.10 Gm.

*Chlorides and Sulphates*: Boil 1 Gm. with 50 mls of water for five minutes, replacing the water lost by evaporation, then filter. 10 mls of the filtrate should give only the faintest turbidity on acidulating with hydrochloric acid and adding 1 mil barium chloride solution or acidulating with nitric acid and adding 1 mil silver nitrate solution.

*Iron and Copper*: Boil 1 Gm. with 45 mls of water and 5 mls hydrochloric acid for five minutes, replacing the water lost by evaporation, and filter. The filtrate should be colourless, and should show no blue colour on the addition of a slight excess of ammonia solution, and no black precipitate, or only the slightest darkening, on the addition of 2 mls of ammonium sulphide solution.

#### Trade Varieties.

Crude animal charcoal or bone black contains about 85 per cent. mineral matter, most of which may be removed by prolonged boiling with dilute hydrochloric acid and subsequent washing. The last portions are exceedingly difficult to remove; even treatment with hydrofluoric acid hardly reduces the proportion of ash below 4 per cent. A good commercial purified animal charcoal contains between 10 and 15 per cent. of ash. It has been stated that purified animal charcoal contains nitrogen firmly combined in an unknown form, and that its decolourising action is proportional to the amount of nitrogen so contained. The decolourising power of various samples is usually tested comparatively on dilute solutions of caramel.

## CHLOROFORM.



Colourless transparent fluid, with a sweet odour and burning taste. Soluble in water 1 in 200, freely miscible with absolute alcohol, ether, benzene, and oils. Sp. gr. 1.5, but when quite

pure it is very unstable, and a trace of alcohol is always added as a preservative.  $\frac{1}{2}$  to 1 per cent. of alcohol is usually employed, and this lowers the sp. gr. to 1.495 to 1.490. Boiling point  $61.5^{\circ}$ .

#### Tests.

*Residue:* 10 mls allowed to evaporate in a clean glass beaker, or dish, which is afterwards heated on a water-bath, should leave no visible residue.

*Smell Test:* If 10 mls be poured upon a circular white filter paper about 10 Cm. in diameter, lying in a flat plate, and allowed to evaporate, no foreign odour should become perceptible, as the last portions disappear, and the filter paper should finally be left quite odourless. This test is usually sufficient to detect impurities occurring through insufficient purification or careless storage.

*Free Acid and Chlorine:* Agitate 5 mls with 10 mls of water. Allow the undissolved chloroform to subside and decant the upper aqueous layer. This should not redden blue litmus paper or give any turbidity on the addition of silver nitrate solution, and if to another portion a few drops of solution of zinc or cadmium iodide, and then starch mucilage, be added, no blue colour should appear.

#### Trade Varieties.

Commercial chloroform is usually very pure. Three varieties are chiefly met with in trade, prepared respectively from duty-paid rectified spirit, methylated spirit, and acetone. When carefully purified the three varieties are undistinguishable by chemical tests, although the varieties prepared from alcohol are said to contain traces of ethyl chloride. Other varieties in less demand are chloroform from chloral hydrate, which is naturally very expensive, and Pictet's chloroform, prepared by exposing pure chloroform to a low temperature and removing the crystals of frozen chloroform from the surrounding fluid.

## CHROMIC ANHYDRIDE.



Commonly known as chromic acid. Dark red needle crystals, very soluble in water, and easily reduced by contact with organic matter. When heated, oxygen is evolved, and a green residue of chromic oxide  $\text{Cr}_2\text{O}_3$  is left. Pure chromic anhydride is much less hygroscopic than impure varieties containing sulphuric acid.

#### Tests.

*Sulphate:* 1 Gm. in 60 mls of water should yield a clear solution, which shows no turbidity after adding 1 ml hydrochloric acid and 5 mls barium chloride solution.





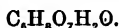


**Potassium Salt:** Carefully heat 1 Gm. in a porcelain crucible to low redness. Triturate the residual chromic oxide with 20 mls of water, filter and evaporate the filtrate to dryness, and dry at 120°-150°. The residue should not weigh more than 0.01 Gm.

#### Trade Varieties.

The chromic acid complying with the tests above is sold as "free sulphate." The article described as "chromic acid, pure," usually contains 90 per cent. or more of  $\text{CrO}_3$ , the remainder being sulphuric acid, potassium sulphate and bichromate, and water. Crude or commercial chromic acid, used for batteries, usually occurs in granular crystalline powder or masses, and contains 60-80 per cent.  $\text{CrO}_3$ , with free sulphuric acid and sodium or potassium sulphates and chromates, besides water.

## CITRIC ACID.



Large colourless prismatic crystals, very soluble in water and alcohol. It is very sensitive to atmospheric changes; it deliquesces in moist and effloresces in dry air. The fully hydrated acid melts at 70°-75° when quickly raised to that temperature, while the anhydrous acid melts at about 150°.

#### Tests.

**Ash:** 1 Gm. carefully ignited should leave no visible residue.

**Oxalic and Tartaric Acids:** 1 Gm. dissolved in 5 mls of a 33 per cent. clear solution of potassium acetate should give no turbidity after standing thirty minutes, even after the addition of an equal volume of 90 per cent. alcohol.

**Sulphuric Acid:** 1 Gm. in 10 mls of water should give no turbidity after the addition of 1 ml barium chloride solution, and setting aside five minutes.

**Lead:** 5 Gm. dissolved in 10 mls of water, nearly neutralised with solution of ammonia, should show no darkening on the addition of 40 mls hydrogen sulphide solution, and setting aside for thirty minutes.

**Iron:** The colourless and faintly acid liquid obtained in testing for lead should show only the faintest darkening on adding sufficient ammonia solution to give the liquid a distinctly alkaline reaction.

#### Trade Varieties.

Citric acid enters into the composition of many articles of diet; hence the purity of commercial specimens is usually satisfactory, as it is so often the subject of analysis. Good trade specimens may contain less than 4 parts of lead per million.

## COPPER.

Copper, in the form of wire, foil, turnings, spinnings, or filings, is used as a reagent in many chemical operations, and in most cases the products derived from ordinary commercial metal are sufficiently pure. When a very pure form of the metal is desired the foil prepared from electrolytic copper, as described below, is usually employed.

### COPPER FOIL, ELECTROLYTIC.

#### Tests.

*Foreign Metals:* Dissolve 4 Gm. in nitric acid diluted with twice its volume of water. Expel the excess of nitric acid by concentrating to a small bulk, and dissolve the product in sufficient water to measure 40 mls. Divide the resulting liquid, which should contain no insoluble particles, into four equal parts.

*Tin:* Evaporate one portion to dryness on a water bath; the residue should be completely soluble in 20 mls of water acidulated with nitric acid.

*Silver:* Evaporate the second portion nearly to dryness, add 50 mls of water and 1 ml hydrochloric acid; no turbidity should appear.

*Lead:* To the third portion add 2 mls sulphuric acid, and evaporate cautiously until the nitric acid is expelled; then dilute with water, when a clear liquid should result.

*Iron:* To the fourth part add 30 mls of water and then solution of ammonia until a clear blue fluid is obtained, smelling strongly of ammonia. Digest for twenty minutes, filter through a white filter-paper, and wash the filter with dilute ammonia until the blue solution is completely removed. Any iron will appear as a reddish stain on the filter-paper.

*Arsenic:* Dissolve 5 Gm. of the copper in nitric acid as before, add 7.5 mls sulphuric acid, and evaporate on a sand bath until white fumes are evolved. Cool and dissolve the residue in 50 mls of water; the resulting liquid may be tested by the Marsh-Berzelius method, and should show no deposit of arsenic in the tube after thirty minutes' treatment.

### CUPRIC AMMONIUM CHLORIDE.



Blue crystals, soluble in water, forming a solution having a slightly acid reaction.

#### Tests.

*Solubility:* 1 Gm. with 10 mls of water forms a bright solution, which should show no deposit after standing twelve hours.

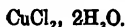




*Iron*: Dissolve 2 Gm. in 100 mls of water, add 5 mls nitric acid, warm for five minutes, and then add solution of ammonia in excess until a clear deep-blue fluid is obtained. Digest twenty minutes, filter through a white filter-paper, and wash the filter with diluted ammonia solution until the blue solution is entirely removed. The paper should show no reddish stain of ferric hydroxide.

*Sulphates*: Dissolve 1 Gm. in 20 mls of water, add 2 mls of barium chloride solution, warm, and set aside. No turbidity should appear within twenty minutes.

## CUPRIC CHLORIDE.



Small green or greenish-blue crystals. Deliquescent, and very soluble in water.

### Tests.

*Solubility*: 1 Gm. should dissolve completely in 10 mls of water or 90 per cent. alcohol, and the solutions should show no deposit after standing twelve hours.

*Sulphate*: 1 Gm. dissolved in 20 mls of water should show no turbidity after adding 2 mls barium chloride solution and setting aside for twenty minutes.

*Iron*: 3 Gm., treated as described under copper ammonium chloride, should show no iron.

*Arsenic*: A solution of 1 Gm. in 20 mls of water should show no indication of arsenical deposit when tested by the Marsh-Berzelius method, the test being continued thirty minutes after the addition of the copper solution. The solution should be added cautiously, as the copper precipitated in the apparatus markedly accelerates the evolution of hydrogen.

*Lead*: 1 Gm. dissolved in 20 mls of water and 2 mls dilute sulphuric acid added should show no turbidity. After standing thirty minutes the liquid should be carefully decanted in order to detect any deposit which may form on the bottom of the test-glass.

### Trade Varieties.

Cupric chloride used for technical purposes contains more or less sulphate and iron, besides being incompletely soluble in water and alcohol.

## CUPROUS CHLORIDE.



A white crystalline powder when freshly prepared, rapidly oxidising when exposed to air, particularly in presence of light and moisture, so that a tinge of green is unavoidable unless the salt is stored in hermetically sealed vessels. Its

value as a reagent is depreciated in proportion to the amount of cupric salt formed, so that samples showing more than a faint green tinge should be rejected. It is insoluble in water, but entirely soluble in ammonia or hydrochloric acid. The ammoniacal solution rapidly oxidises, but the hydrochloric acid solution, which is brownish-black, may be completely reduced and rendered almost colourless by digestion with copper in the form of wire, foil, or shavings.

#### Trade Varieties.

Cuprous chloride for technical purposes is sold as a powder, which is greenish from partial oxidation, and when freshly made usually has a brown colour, due chiefly to the high temperature at which the precipitated cuprous chloride, often incompletely washed, has been dried.

## CUPRIC OXIDE.

$\text{CuO}$ .

Cupric oxide is used in several different physical forms, which are prepared as described below:—

1. "Precipitated powder," made by precipitating a solution of cupric sulphate with sodium hydroxide, boiling the liquid, and collecting and washing the precipitated hydrated oxide. This is then dried and ignited to expel the combined water.

2. "Granulated," made by heating the precipitated oxide to incipient fusion, and stirring the semi-molten mass. After cooling, the product is sifted to separate the granules of suitable size.

3. "From wire." This is made by heating bundles of copper wire to low redness in a current of air. Wire of No. 16 to 26 B.W.G. is usually employed; with thicker wire difficulty is experienced in oxidising the wire throughout, and in some pieces a small core of unoxidised copper may be found.

Cupric oxide is very hygroscopic, and must always be freshly ignited when used for elementary analysis. Cupric oxide obtained by heating the nitrate is not often employed owing to the difficulty of removing the last traces of nitric compounds. Moreover, such oxide usually contains heavy traces of iron, since the final ignition is carried out in iron pots. The oxide may also be obtained by igniting the carbonate.

#### Tests.

**Nitrate:** Heat 10 Gm. in a dry test-tube to low redness. No nitrous fumes should be evolved, and no change should be produced in a piece of moistened blue litmus paper carefully lowered into the tube just above the oxide.

**Carbonate:** Place 20 Gm. in a Jena flask heated by a Bunsen burner and aspirate air through the flask (first passing







the air through a wash-bottle containing strong potash solution to remove carbon dioxide) and then through a vessel containing lime water; no turbidity should be produced in the lime water.

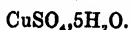
*Sulphate*: Dissolve 0.5 Gm. in 3 mls of hydrochloric acid, dilute the solution to 25 mls, and add 2 mls barium chloride solution; no turbidity should be produced after setting aside two hours.

*Iron and Calcium*: Dissolve 1 Gm. in 5 mls of hydrochloric acid, dilute to 50 mls, and pass hydrogen sulphide through the solution until the copper is entirely precipitated. The filtrate, evaporated to dryness and the dish gently heated, should leave a residue weighing less than 0.01 Gm. This may be tested for iron and calcium.

#### Trade Varieties.

- Commercial black oxide of copper often contains heavy traces of sulphates if made by precipitation from the sulphate. When obtained as a by-product in metallurgical operations, or by igniting copper scrap or scales, considerable quantities of cuprous oxide or metallic copper may be present as well as iron and calcium compounds.

## CUPRIC SULPHATE.



Blue, translucent crystals, soluble in  $3\frac{1}{2}$  parts of water, almost insoluble in alcohol. The aqueous solution has an acid reaction to litmus paper.

#### Tests.

*Solubility*: 2 Gm. dissolved in 20 mls of water should form a perfectly clear solution, which should show no deposit on standing for twelve hours.

*Iron*: 4 Gm. dissolved in 100 mls of water and tested as described under cupric ammonium chloride should show no trace of iron.

*Other Impurities*: 4 Gm. are dissolved in 100 mls of water, 2 mls of hydrochloric acid added, and the copper completely precipitated with hydrogen sulphide. The liquid should eventually be heated nearly to boiling while the gas is passing through it. The filtrate, after evaporation, should leave no residue, or one weighing not more than 0.002 Gm. after ignition.

#### Trade Varieties.

Crude copper sulphate, known as blue copperas, is used for agricultural purposes, and has usually a greenish colour from presence of considerable quantities of ferrous sulphate. Commercial copper sulphate, sold as 98 to 99 per cent. pure,

occurs in large blue crystals, and is not completely soluble in water, besides containing considerable traces of iron. "Recrystallised" copper sulphate is usually nearly pure,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , and contains only small traces of iron, which cannot easily be entirely removed by simple recrystallisation from water.

## ETHER.



A colourless, transparent, mobile fluid, with a penetrating odour. Boiling point,  $35^\circ \text{C}$ .; sp. gr., 0.720.

### Tests.

*Residue:* 20 mls allowed to evaporate from a glass dish and the dish subsequently warmed on a water-bath should leave no residue. Before warming the dish a film of moisture will be found in it, due to moisture attracted from the air by the ether during evaporation.

*"Smell" Test:* Pour 20 mls over a circular filter paper resting in a clean plate, and allow the ether to evaporate. No foreign odour should be detected at any stage of the evaporation, and when the odour of ether is no longer perceptible the paper should be quite odourless. This empirical test is very effectual in detecting such unidentifiable impurities as result from imperfect purification.

*Free Acid:* Shake the ether with an equal volume of water and drop in a piece of blue litmus paper and shake again. The colour should not be altered or discharged.

*Water:* Ether should form a clear liquid when added to an equal volume of carbon bisulphide. This test detects the presence of more than traces of water.

*Aldehyde:* Add 5 Gm. potassium hydroxide in stick form to 20 mls of ether in a stoppered bottle, shake, and set aside for two hours. No discolouration of the liquid or stick should occur.

*Peroxides:* Dissolve about 0.1 Gm. potassium chromate in 9 mls of water, add 1 mil of dilute sulphuric acid, and shake with 2 mls of ether. After separation the ether should be colourless. If peroxides be present perchromic acid is formed, which dissolves in the ether, imparting to it a blue colour. Another test is to agitate the ether with one-tenth its volume of potassium iodide solution and set aside for ten minutes, when no brown colour should be apparent. In order to comply with either of these tests the ether must have been kept from the light, as a very short exposure after purification in manufacture is sufficient to develop traces of peroxides.





**DRY ETHER, DISTILLED OVER SODIUM.**

This is prepared by adding shavings of sodium to pure ether, contained in a flask, so long as bubbles of gas are evolved. The flask is then connected to a condenser and receiver, provided with drying tubes to prevent access of moisture from the atmosphere, and distilled. The distillate when treated with fresh sodium cuttings should not readily tarnish the fresh metallic surface, and only a few small bubbles of gas should be evolved.

**Trade Varieties.**

Besides pure ether, as described above, numerous qualities and varieties are sold. These fall into two groups—one prepared from duty-paid rectified spirit (ether from pure spirit) and the other from industrial alcohol, which is exempt from duty, and contains 5 per cent. of wood naphtha (ether from methylated spirit). The presence of the methyl alcohol of the wood naphtha in the industrial alcohol causes the ether derived therefrom to contain methyl oxide and methyl-ethyl oxide. These have a lower boiling point and sp. gr. than ethyl oxide, hence "methylated" ethers commence to boil below  $35^{\circ}$  C. and have a lower sp. gr. than the corresponding product from pure alcohol. The variations in the sp. gr. of commercial ethers is also caused by the presence of varying proportions of alcohol and water. The following are the chief qualities usually sold:—

**Ether Pure 0.720.**—Pure ether from rectified spirit. Nearly free from water.

**Ether Pure 0.735.**—Ether from rectified spirit containing nearly 10 per cent. water and alcohol. This should comply with the smell test, but contains aldehyde and peroxides.

**Ether Meth. 0.717.**—Nearly anhydrous, but contains methyl oxide. Commences to boil at about  $20^{\circ}$  C., and mostly used as a spray for local anæsthesia.

**Ether Meth. 0.720.**—Contains varying proportions of alcohol and water, and sometimes complies with the carbon bisulphide test for water. Contains aldehyde and peroxides, but should give negative results with the "smell" test. It is sufficiently good for most laboratory uses.

**Ether Meth. Purif. 0.720.**—Usually complies with all the tests given above, except that for peroxides, and the boiling point is somewhat wide,  $33^{\circ}$ - $36^{\circ}$  C.

**Ether Meth. 0.730.**—This contains much water and alcohol, and is used only as a solvent for technical use.

**Ether Meth. 0.735.**—This is a slightly lower grade than the last-mentioned, and is mostly employed in ice machines.

**GOLD CHLORIDE.**

Gold chloride, or aurochloric acid, the formula being sometimes written  $\text{HAuCl}_4, 4\text{H}_2\text{O}$ , occurs in yellow deliquescent crystalline masses, freely soluble in water and alcohol. The salt, according to the formula, contains 48 per cent. of metallic gold, but if free from impurities a slightly higher percentage will be found, owing to the slight loss of hydrochloric acid and water which occurs in drying the crystals.

**Tests.**

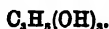
*Percentage of Gold:* Dissolve 0.5 Gm. in 25 mls of water, render the solution distinctly alkaline with solution of potassium hydroxide, add 5 mls of hydrogen peroxide solution (10 vols.), and heat to about  $100^\circ$  for an hour. Collect the precipitated gold, wash it with water acidulated with hydrochloric acid, dry, and cautiously ignite; the residue should weigh not less than 0.24 Gm.

*Foreign Metals:* The filtrate from the foregoing test should not be affected by passing hydrogen sulphide through it, or by ammonium sulphide solution after supersaturation with ammonia.

*Sodium Salt:* Cautiously ignite 0.2 Gm. of the salt mixed with 0.5 Gm. oxalic acid in a porcelain crucible until all volatile matter has been driven off. Cool, and triturate the residue with 10 mls of water, filter, evaporate the filtrate to dryness. The residue should weigh less than 1 Mgm.

**Trade Varieties.**

Sodium aurochloride ( $\text{NaAuCl}_4, 2\text{H}_2\text{O}$ ), containing 49.5 per cent. of metallic gold, is sometimes sold as gold chloride, especially for photographic use. It is not so deliquescent as the foregoing salt, and is more easily obtained in detached yellow needle crystals. For analytical use the presence of the sodium is sometimes objectionable. Many varieties of so-called gold and sodium chloride are not the true compound, but contain only 20 to 30 per cent. metallic gold, and consist of gold chloride mixed with more or less sodium chloride.

**GLYCERIN.**

Viscid, clear, colourless, and odourless fluid, with a sweet taste. Miscible in all proportions with alcohol and water, but insoluble in chloroform, ether, benzene, or petroleum ether. Sp. gr., 1.25 to 1.26. Boiling-point, when anhydrous,  $290^\circ$  C. Readily attracts moisture when exposed to the air.







**Tests.**

*Residue:* 5 mls heated in a dish gives off inflammable vapours and leaves only a black stain, which should entirely disappear when heated to low redness.

*Reaction:* A 10 per cent. solution in water should be quite neutral to red or blue litmus paper.

*Chlorides, Sulphates, or Oxalates:* A 1 in 10 solution in water should give no turbidity on the addition, to separate portions, of one-fifth its volume of silver nitrate or barium chloride solution and setting aside thirty minutes.

*Metals:* A 1 in 10 solution in water should show no darkening when treated with hydrogen sulphide to saturation, or on the addition of one-twentieth its volume of ammonium sulphide solution.

*Fatty Acids:* 10 mls mixed with 5 mls of alcohol and 5 mls of dilute sulphuric acid, and the mixture warmed by immersing the tube in boiling water, should give no fruity odour.

*Cane and Grape Sugars:* Place 1 mil of glycerin with 20 mls of water in a small flask, add 1 drop strong hydrochloric acid, and heat the flask on the water-bath for thirty minutes. Then add 2 mls potassium hydroxide solution and 2 mls of Fehling's solution, heat to boiling, and set aside to cool. No red precipitate should appear.

*Foreign Organic Matter:* (a) To 5 mls in a test-tube add gradually 5 mls sulphuric acid, keeping the tube cool during the addition of the acid. Set aside for one hour; the mixture should remain colourless, or acquire at most a pale straw colour. (b) Mix 5 mls with 5 mls of ammonia solution, add 0.5 mil silver nitrate solution, and shake. No darkening in colour should result.

*Ammonium Salts:* Mix 5 mls with an equal volume of water, add 1 mil potassium hydroxide solution, and heat nearly to boiling. A piece of moistened red litmus suspended in the vapour above the liquid should not be turned blue.

*Arsenic:* Tested by the Marsh-Berzelius method, it should show less than 1 in 250,000 of arsenic.

**Trade Varieties.**

Glycerin for technical purposes is sold in various qualities, in colour from brown to nearly water-white. The sp. gr. is usually lower than 1.25, while any of the foregoing impurities may be present even in colourless samples.

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**HYDRIODIC ACID.**

A colourless gas, which dissolves freely in water, a solution saturated at 0° C. having a sp. gr. of about 2, and containing 90 per cent. HI. Strong solutions rapidly become coloured from liberation of free iodine by oxidation, but this change

does not usually interfere with the use of the acid as a reducing agent. It is usually sold in two strengths:—

(a) Sp. gr. 1.5, containing 43.45 per cent. HI.

(b) Sp. gr. 1.68-1.72, containing 51.53 per cent. HI. This strength is employed in Zeisel's method for the determination of methoxyl groups. For reagent purposes the acid is best prepared by the iodine and phosphorus method, as it is very difficult to remove traces of sulphur compounds when the hydrogen sulphide method is employed.

#### Tests.

*Residue*: 2 mls evaporated in a capsule and the dish gently heated should leave less than 0.2 Mgm. residue.

*Sulphates*: 1 mil diluted to 20 mls with water, and 2 mls barium chloride added, should show no turbidity after standing three hours.

*Phosphates*: 1 mil diluted to 20 mls with water, and rendered slightly alkaline by the addition of ammonia solution, should yield no turbidity on the addition of 2 mls ammonio-magnesian reagent (magnesia-mixture) and setting aside three hours.

*Chloride and Bromide*: Dilute 1 mil to 20 mls with water, add silver nitrate solution in excess, which is indicated by the rapid setting of the flocculent precipitate. Decant the supernatant fluid, add 20 mls ammonia solution, shake well, and filter rapidly. To the filtrate add nitric acid until it has a faint but distinctly acid reaction. The fluid should only show a faint opalescence.

#### Trade Varieties.

Concentrated hydriodic acid is usually pure.\* Sulphate may be found if prepared by the hydrogen sulphide process. A 10 per cent. acid, used in medicine, and made by mixing concentrated solutions of potassium iodide and tartaric acid, is also met with. This leaves a residue of 1 per cent. or more of potassium salt on evaporation, and often contains phosphate, owing to the addition of hypophosphorous acid for removing traces of free iodine.

## HYDROBROMIC ACID.

A dense, colourless gas, which fumes in the air and dissolves freely in water. The solutions most commonly used as reagents are as follows:—

Sp. gr. 1.209 = 25 per cent. HBr.

Sp. gr. 1.260 = 30 per cent. HBr.

Sp. gr. 1.375 = 40 per cent. HBr.

Sp. gr. 1.450 = 45 per cent. HBr.

The stronger solutions usually show a yellow tint from the liberation of bromine which occurs on keeping.





**Tests.**

*Residue:* 5 mils evaporated in a capsule, which is finally heated on a sand-bath, should leave no visible residue or one weighing less than 0.5 Mgm.

*Sulphates:* 1 mil diluted to 10 mils with water and 2 mils barium chloride solution added should show no turbidity after standing three hours.

*Phosphates:* 1 mil diluted with 20 mils of water and rendered slightly alkaline by the addition of ammonia solution should yield no turbidity on the addition of 2 mils ammonio-magnesian reagent (magnesia mixture), and setting aside three hours.

*Metals:* A 1 in 10 dilution of the acid should show no darkening when mixed with twice its volume of hydrogen sulphide solution, or after rendering alkaline with slight excess of ammonia solution, a few drops of ammonium sulphide solution, and setting aside one hour.

**Trade Varieties.**

These usually show more than traces of sulphate or phosphate and a residue exceeding that given above. For medicinal use a 10 per cent. acid made by mixing strong solutions of tartaric acid and potassium bromide may be employed; this is unsuitable for reagent use.

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**HYDROCHLORIC ACID.**

A dense, colourless gas, dissolving freely in water. The solution most commonly sold as a reagent has a sp. gr. of 1.16, and contains 31.8 per cent. HCl. It should be clear and colourless.

**Tests.**

*Residue:* 50 mils evaporated in a capsule which is finally heated on a sand-bath should leave no visible residue or one weighing less than 1 Mgm.

*Sulphates:* 100 mils are evaporated to 5 mils and then diluted with water to 100 mils. On adding 5 mils barium chloride solution and setting aside twelve hours only a faint deposit of barium sulphate at most should be formed, and if this be collected it should weigh less than 0.4 Mgm., corresponding to about 0.00017 per cent.  $H_2SO_4$ . In detecting small traces of sulphuric acid the preliminary evaporation is necessary in order to remove most of the hydrochloric acid. Another plan, which is quicker, but not so reliable, is to nearly neutralise with the purest sodium carbonate after dilution, and then add the barium chloride.

*Arsenic:* 25 mils of the acid treated in the Marsh-Berzelius apparatus for thirty minutes should show only a faint arsenical deposit, corresponding to less than 1 part per million of

arsenic. Freshly prepared samples of acid which pass this test will frequently be found to contain more arsenic after keeping even a few weeks, the arsenic being derived from the glass bottles. Some observers state that they find the bottles in which bromine is imported to be least liable to contaminate hydrochloric acid, but whether this may be referred to the composition of the glass or to any arsenic present in the glass having been extracted by the bromine has not been determined.

*Metals:* Dilute 10 mls to 50 mls with water, and add an equal volume of hydrogen sulphide solution. No darkening should occur after standing one hour.

*Iron:* Dilute 2 mls to 20 mls with water, and add 1 mil potassium thiocyanate solution. No pink colouration should appear.

*Alkalies and Alkaline Earths:* These are excluded by the stringent test for residue on evaporation.

*Free Chlorine:* Dilute 2 mls to 20 mls with water, add 1 mil of potassium iodide solution, and then 5 mls of starch solution. No blue colouration should be immediately produced.

#### Trade Varieties.

The "pure" hydrochloric acid of commerce will not usually pass the stringent tests given above, particularly those relating to residue and sulphate. Crude yellow hydrochloric acid or "spirit of salts" contains iron and gives a considerable residue on evaporation. Arsenic is present in considerable quantity, while some samples contain several parts per hundred of sulphuric acid.

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## HYDROFLUORIC ACID.

A colourless gas, with a very irritating odour, soluble in water, forming a solution which has a very corrosive action on the skin and must be handled with great care. The solution sold as a reagent contains 40 per cent. HF. It is usually stored in gutta-percha bottles closed by a rubber cork, but even these materials are slowly attacked by hydrofluoric acid, hence it is better to employ bottles made of hard paraffin wax or ceresine.

#### • Tests.

*Residue:* 10 grammes evaporated in a platinum basin should yield no residue or one weighing less than 0.5 Mgm. after ignition at a low red heat. If the acid has been stored in gutta-percha bottles a slight dark film may be found in the basin, which is mostly dissipated by heating to low redness, leaving a minute dark residue consisting partly of iron.

*Alkalies and Alkaline Earths:* These are sufficiently excluded by the residue test.







*Sulphate*: 1\*gramme evaporated to complete dryness in a platinum dish over the water bath, and the dish rinsed with 10 mils of water, should yield a liquid which gives no immediate turbidity on the addition of two drops of hydrochloric acid and 1 mil of barium chloride solution. It is necessary to remove the hydrofluoric acid by evaporation, owing to the insolubility of barium fluoride.

*Silica*: Dilute 2 grammes with 10 mils of water, and add a solution of 1 gramme of potassium chloride in 5 mils of water. No turbidity or precipitate of potassium silicofluoride should be produced.

*Metals*: Dilute 5 grammes with 40 mils of water, and add 50 mils hydrogen sulphide solution. No precipitate or darkening should occur.

*Iron*: Pour 2 grammes into 20 mils of water, add sufficient solution of ammonia (about 10 mils) to give a distinctly alkaline reaction, and then 5 drops ammonium sulphide solution. No darkening should ensue after standing five minutes.

#### Trade Varieties.

Hydrofluoric acid is put on the market of very varying purity. The better qualities for technical purposes leave very little residue on evaporation, but many are dark-coloured, and contain lead and considerable quantities of silica and sulphuric acid. The total acidity of such crude acids cannot therefore be taken as a measure of the hydrofluoric acid present. It should be noted that absence of residue on evaporation is no indication of the absence of silica, since hydrofluosilicic acid is volatile.

## HYDROGEN PEROXIDE.

A solution of hydrogen peroxide in water. It is sold in several degrees of concentration, which are usually described by the number of volumes of oxygen which one volume of the solution will yield. In the gasometric determination with potassium permanganate it must be remembered that half the volume of oxygen actually obtained is derived from the permanganate, so that 1 mil of a 10-volume solution should give 20 mils of oxygen. The following strengths are usually sold:—

- 10 volumes equivalent to nearly 3 per cent.  $\text{H}_2\text{O}_2$ .
- 20\* volumes equivalent to nearly 6 per cent.  $\text{H}_2\text{O}_2$ .
- 30 volumes equivalent to nearly 9 per cent.  $\text{H}_2\text{O}_2$ .
- 40 volumes equivalent to nearly 12 per cent.  $\text{H}_2\text{O}_2$ .
- 100 volumes equivalent to nearly 30 per cent.  $\text{H}_2\text{O}_2$ .

The weaker solutions are best kept in bottles closed by corks which have a piece of cane passing through the centre, while

the 30 per cent. is usually sent out in bottles coated internally with paraffin wax.

### Tests.

For the qualitative tests described below the solution should be diluted with distilled water, so that the liquid contains about 1 per cent. hydrogen peroxide.

*Reaction*: Faintly acid to blue litmus paper. After boiling with a trace of lead peroxide to decompose the hydrogen peroxide the reaction should be neutral.

*Residue*: 50 mils of a 10-volume solution, or the equivalent volume of the stronger solutions, evaporated on a water-bath, should leave less than 1 Mgm. residue. This test excludes the presence of more than minute traces of sodium, magnesium, or barium salts.

*Sulphates*: To 20 mils add 1 mil hydrochloric acid, boil for one minute, add 1 mil barium chloride solution. No turbidity should appear after standing two hours.

*Phosphates*: To 10 mils add 2 mils of 10 per cent. ammonia solution and 2 mils of magnesium mixture. No turbidity should appear after standing twelve hours.

*Chlorides*: 20 mils with 1 mil of nitric acid and 1 mil of silver nitrate solution should remain free from turbidity after standing two hours.

*Quantitative Determination*: This may be carried out by titration with N/10 potassium permanganate in acid solution, or by adding the liquid to a solution of potassium iodide acidified with sulphuric acid and titrating the liberated iodine with sodium thiosulphate.

### Trade Varieties.

Commercial hydrogen peroxide is used for many technical purposes in varying degrees of purity. Usually considerable traces of free acid are present, and a residue amounting to 0.5 per cent. may be obtained. Barium is also found in some samples. The addition of ether or alcohol is said to act as a preservative.

## HYDROXYLAMINE HYDROCHLORIDE.



Clear, colourless crystals, soluble in water and alcohol.

### Tests.

*Residue*: 1 gramme gently heated in a crucible should leave less than 0.5 Mgm. residue.

*Ammonium Salts*: A solution of 1 gramme in 20 mils 90 per cent. alcohol to which 1 mil platinum perchloride solution is added should remain free from turbidity after standing one hour





**IODINE.**

Large greyish-black scales, with a metallic lustre and characteristic odour. Soluble in alcohol, ether, chloroform, and carbon bisulphide; less soluble in glycerin, sparingly soluble in water (1 in 7,000), but freely soluble in solutions of potassium iodide.

**Tests.**

*Residue:* 1 gramme gently heated in a white porcelain dish should leave no visible residue.

*Moisture:* The solution in chloroform should be transparent, and the scales shaken in a dry bottle should not adhere to the sides.

*Quantitative Determination:* Titration with N/10 sodium thiosulphate should indicate 99.9 to 100 per cent. iodine. If a higher result be obtained probably traces of bromine or chlorine are present.

**Trade Varieties.**

The iodine of commerce in masses is not suitable for reagent use. It is not entirely soluble in the solvents mentioned above, and leaves more or less residue on evaporation.

**IODIC ACID.**

Colourless crystals, easily and entirely soluble in water; nearly insoluble in absolute alcohol or ether.

**Tests.**

*Residue:* 1 gramme heated to low redness in a crucible leaves a residue weighing not more than 0.5 Mgm.

*Quantitative Determination:* This is carried out by adding the salt to a solution of potassium iodide acidulated with sulphuric acid, and titrating the liberated iodine with N/10 sodium thiosulphate.

**IODIC ANHYDRIDE.**

Colourless crystalline powder, easily and entirely soluble in water, forming a solution of iodic acid. It should comply with the tests given under Iodic Acid.

**IRON.**

(1) Metallic iron in the form of filings, borings, and turnings is used as a reducing agent. For most purposes any of the commercial varieties of iron are sufficiently uniform and good. For some purposes a special form of powder obtained

by cleansing and grinding iron filings is employed. This is known as "levigated" iron filings, or "*ferrum pulveratum*," and should contain 98 per cent. metallic iron when tested by the copper sulphate method, described under "Reduced Iron."

(2) Reduced Iron.—This is a dull black powder containing metallic iron and some oxides of iron, obtained by reducing iron oxide with hydrogen at a low red heat. When examined with a lens it should not exhibit any angular glistening fragments.

### Tests.

*Percentage of Metallic Iron:* Several methods are used for this, but while concordant results may be obtained from different samples by each method, the results of the various methods are not concordant on the same sample. The iodine and mercuric chloride method agree fairly well, but higher results are given by the copper sulphate method when carried out as described below. The details are as follow :—

*Copper Sulphate Process.*—Dissolve 5 Gm. copper sulphate in 60 mils freshly boiled and cooled distilled water contained in a 100-mil flask furnished with a Bunsen valve. Finely powder the reduced iron and introduce 1 Gm. into the copper sulphate solution. Shake the flask well, heat the contents to boiling, and keep on a water-bath for thirty minutes, shaking at intervals. Cool quickly, and make up the contents to 100 mils with freshly boiled water. Filter and take 10 mils of the filtrate, add 10 mils dilute sulphuric acid, and titrate with N/10 potassium permanganate, of which not less than 16 mils should be required to give the usual permanent pink tint. This is a modification of the British Pharmacopœia method. 1 mil N/10  $\text{KMnO}_4$  solution = 0.0056 Gm. iron.

*Residue Insoluble in Acid.*—1 Gm. gently warmed with 20 mils dilute hydrochloric acid until action ceases should leave a small residue which, when collected on a filter, washed, and ignited, weighs not more than 0.01 Gm.

*Arsenic.*—Direct examination in a Marsh apparatus gives negative results or results far below the figure corresponding to the arsenic present, because the arsenic appears to exist in the form of an iron arsenide which does not readily react with nascent hydrogen. It is necessary to treat the reduced iron as follows :—Heat 0.5 Gm. with 1 Gm. potassium chlorate and 10 mils strong hydrochloric acid until the evolution of chlorine ceases. Evaporate the fluid to dryness on a water-bath, redissolve the residue in water, and introduce the fluid into a Marsh-Berzelius apparatus. The arsenical mirror then obtained should not exceed that corresponding to 0.05 Mgm. of arsenic, or less than 1 in 10,000 in the weight of reduced iron taken.







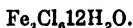
**Heavy Metals.**—The filtrate obtained in testing the reduced iron for insoluble residue is heated with 5 mls strong nitric acid, then diluted with 25 mls water, and a slight excess of ammonia added. Filter out the precipitated ferric hydroxide; the filtrate should be colourless, and should not be darkened when mixed with an equal volume of hydrogen sulphide solution, and set aside for one hour.

#### Trade Varieties.

Reduced iron can be purchased containing any percentage of iron in metallic form from 50 to 90 per cent. The British Pharmacopœia only requires 75 per cent., while the German and United States Pharmacopœias specify a product containing 90 per cent. iron.

(3) **Iron Wire.**—Clean bright piano wire is used for standardising volumetric solution of potassium permanganate. It should contain 99·6 to 99·9 per cent. metallic

## IRON CHLORIDE (FERRIC).



Yellow or orange-yellow masses with a crystalline fracture rapidly deliquescent in air. Easily soluble in water and alcohol, giving an acid reaction.

#### Tests.

**Solubility:** The salt should dissolve, forming a clear solution in its own weight of distilled water.

**Zinc, Copper, and Alkalies:** Dissolve 5 Gm. in 100 mls of water. Completely precipitate with the addition of ammonia solution in slight excess, boil and filter. The filtrate should be colourless, and one half evaporated to dryness and ignited should leave a residue not exceeding 1 Mgm. A portion of the filtrate should give no precipitate with ammonium sulphide solution.

**Nitrates:** 1 Gm. is dissolved in 10 mls of water and completely precipitated by the addition of ammonia and filtered. Neutralise the filtrate, add a crystal of ferrous sulphate, and carefully pour sulphuric acid down the side of the tube without mixing the liquids; no brown zone should appear at the junction of the two fluids.

**Ferrous Salts:** A 2 per cent. solution should give no blue colour on the addition of a few drops of potassium ferri-cyanide solution.

**Sulphate:** 1 Gm. dissolved in 50 mls of water should give no immediate turbidity on the addition of 5 mls of barium chloride solution.

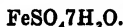
**Arsenic:** Place 2 Gm. in a small retort with 30 mils of hydrochloric acid, 12 mils water, 4 Gm. ferrous sulphate, and 4 Gm. potassium metabisulphite. Distil almost to dryness, and evaporate the distillate in a porcelain dish with 3 mils nitric acid and 5 mils sulphuric acid until the sulphuric acid fumes begin to appear. The residual fluid introduced into the Marsh-Bérzelius apparatus should give only the faintest arsenical stain after thirty minutes.

#### Trade Varieties.

Ferric chloride is made in all grades of purity. The cruder varieties used only for technical purposes are obtained often as by-products, and usually contain noticeable quantities of arsenic, nitric acid, and sulphates. The hydrated compound described above must be distinguished from the anhydrous ferric chloride.

Ferric chloride is also sold in the form of solutions whose strengths are usually determined by their specific gravities. It occurs in this form in several pharmacopœias under the name of *Liquor Ferri Perchloridi*.

## IRON SULPHATE (FERROUS).



Light green crystals easily soluble in water, insoluble in alcohol and ether. The crystals should be bright on the surface and free from any rusty incrustation.

#### Tests.

**Solubility:** 2 Gm. should dissolve to a clear solution in 10 mils freshly boiled water.

**Copper, Zinc, and Alkalies:** Dissolve 5 Gm. in 50 mils of water. Oxidise by the addition of 5 mils of nitric acid and boil. Completely precipitate the iron by a slight addition of ammonia and filter. Divide the filtrate into three parts. One part evaporated to dryness and ignited should leave a residue not exceeding 1 Mgm. To another portion add ammonium sulphide; no discolouration should ensue. The third portion in a white glass tube held above a white surface should show no blue colour.

**Quantitative Determination:** The salt should be dissolved in freshly boiled and cooled distilled water acidulated with sulphuric acid and titrated with N/10 permanganate solution. Good samples should show over 99.5 per cent. ferrous sulphate.

#### Trade Varieties.

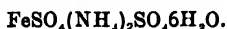
Ferrous sulphate under the name of green vitriol or copperas is sold in very crude forms for technical purposes.





These forms contain copper, often arsenic and zinc, and are usually rusty from partial oxidation. The pure salt is often sold in granules obtained by pouring the concentrated aqueous solution into alcohol which precipitates the salt in fine crystals, having, however, the same composition as the large crystals described above.

## IRON AMMONIUM SULPHATE (FERROUS)



Pale green crystals soluble in six parts of water, insoluble in alcohol. It may be useful to remember that this salt contains one-seventh its weight of metallic iron.

### Tests.

This salt should answer the tests described under ferrous sulphate.

### Trade Varieties.

The salt is often sold in the granular form, which is very convenient for effecting rapid solution, and keeps well.

## IRON SULPHIDE (FERROUS).



Greyish-black fragments with granular fracture, sold in irregular masses, plates of varied thickness, or stick form.

### Tests.

*Solubility:* 0.5 Gm. warmed with 20 mls of water and 5 mls hydrochloric acid until effervescence ceases should entirely dissolve or leave only a residue of less than 0.02 Gm.

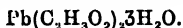
*Quantitative Determination:* This is made by warming with dilute sulphuric acid and passing the evolved gases into N/10 iodine solution, the residual iodine being afterwards determined by means of N/10 sodium thiosulphate. The determination is based upon the reaction  $\text{H}_2\text{S} + 2\text{I} = 2\text{HI} + \text{S}$ .

### Trade Varieties.

A good deal of the inferior sulphide of iron on the market is nothing more than a crude sulphide of iron slag obtained as a by-product, and effervesces with very great difficulty when treated with acid. Bad qualities also often contain considerable quantities of uncombined iron, and these, while effervescing freely with dilute acid, evolve gases containing a large proportion of hydrogen.

**LEAD.**

Metallic lead in the form of foil or granulated is sold containing less than 5 dwts. of silver per ton, and in this form may be regarded as free from silver for analytical purposes. The determination of silver in such small quantities can only be done by cupellation on large quantities.

**LEAD ACETATE.**

Colourless transparent crystals, easily soluble in water, having a sweetish astringent taste, and a faint acetous odour. The salt effloresces and loses acetic acid on exposure to air becoming thereby partly insoluble in water.

**Tests.**

*Solubility:* 5 Gm. should dissolve in 50 mls freshly boiled and cooled distilled water with only the faintest opalescence, and this should immediately disappear on the addition of one drop of acetic acid.

*Copper and Iron:* 0.5 Gm. dissolved in 10 mls of water and treated with potassium ferrocyanide solution should give a pure white precipitate free from any tinge of blue or brown.

*Chloride:* 1 Gm. dissolved in 20 of distilled water with two drops of acetic acid should give no immediate precipitate with silver nitrate solution.

*Alkalies and Alkaline Earths:* Dissolve 2 Gm. in 40 mls of water. Add hydrochloric acid drop by drop until no further precipitate is produced and then remove the remainder of the lead from the solution by hydrogen sulphide and filter. The filtrate evaporated to dryness and ignited should yield a residue weighing not more than 1 Mgm.

**Trade Varieties.**

The commercial salt is largely used as sugar of lead in medicine and the arts. It usually contains basic carbonate due to dissociation in contact with air, and is therefore not completely soluble in freshly boiled water. It also usually contains traces of iron and copper.

**LEAD CHROMATE.**

Heavy yellowish-brown powder or granules of the same produced by partial fusion and stirring.







**Tests.**

*Solubility:* 1 Gm. heated with 10 mls of nitric acid 1.42 should almost entirely dissolve.

*Organic Substances:* No carbonic acid should be evolved on ignition in a stream of oxygen. The best method of testing is to place 10 Gm. of the lead chromate in a combustion tube and treat it as for a blank experiment, passing the stream of oxygen first through potash absorption bulbs.

*Soluble Substances:* 1 Gm. finely powdered is digested in 20 mls of hot water with constant shaking for ten minutes. The filtrate, evaporated to dryness and ignited, should leave a residue of less than 1 Mgm.

**Trade Varieties.**

Lead chromate and oxychromate are used very largely in pigments, and vary in colour from pale yellow to deep orange.

Lead chromate used for ultimate organic analysis must be kept in a well-closed bottle in order to avoid contamination with organic dust.

**LEAD OXIDE.****PbO.**

Yellow to orange powder easily soluble in acetic acid or nitric acid.

**Tests.**

*Metallic Lead and Carbonate:* 5 Gm. are suspended in 15 mls of water, and 20 mls 33 per cent. acetic acid are added without agitation. No bubbles of gas should be evolved, and on gently warming the lead oxide should dissolve, forming a solution which should be clear, or at the most only slightly cloudy. The solution should be filtered and used for the subsequent tests.

*Chlorides:* To 5 mls of the filtrate add an equal bulk of water and 1 ml silver nitrate solution. No precipitate should appear immediately.

*Copper:* To 5 mls of the filtrate add an equal bulk of water and then 5 mls dilute sulphuric acid. Warm, filter, add one drop sulphuric acid to the filtrate to show that all the lead has been precipitated. Add ammonia drop by drop until the liquid becomes alkaline. No blue colour should appear.

*Calcium:* The alkaline fluid from the preceding test should give no precipitate on the addition of ammonium oxalate solution.

**Trade Varieties.**

Lead oxide is largely used under the name of litharge, which is sold in scales or the same ground to powder. In

making litharge the oxidation is so conducted that the oxide itself is fused. Massicot is another variety of lead oxide which is produced by the oxidation of lead at temperatures below the melting point of the oxide. Massicot is much more readily oxidised to red lead than litharge. The hydrated oxide occurs as a white powder, and is produced by precipitating a solution of a lead salt with an alkali hydroxide.

## LEAD PEROXIDE.



A dark brown amorphous powder.

### Tests.

*Solubility:* Insoluble in water, attacked by strong hydrochloric acid with evolution of chlorine, and strong sulphuric acid with evolution of oxygen.

*Soluble Salts:* 1 Gm. boiled with 25 mls of water for two minutes and filtered. The filtrate evaporated to dryness and gently ignited should leave a residue of less than 1 Mgm.

*Manganese:* 1 Gm. is heated with 3 mls strong sulphuric acid until decomposition is complete and a perfectly white residue remains. After cooling this is placed in about 10 mls of water and a few grains of lead peroxide again added. Heat the mixture to boiling and set aside to allow the insoluble portion to completely subside. The supernatant fluid should be quite white and free from any red colour.

### Trade Varieties.

Commercial lead peroxide usually contains considerable quantities of soluble salts, chiefly lead nitrate, which has been left in the product after the action of nitric acid on the red lead from which it is made. Chlorides are also frequently present in samples made by the action of chlorinated soda or lime on a solution of lead salt.

## MAGNESIUM CARBONATE.

This is a hydrato-carbonate of magnesium, containing about three molecules of carbonate to one of hydroxide.

White powder, insoluble in water, easily soluble in acid, with effervescence.

### Tests.

*Solubility:* Suspend 3 Gm., in 50 mls of water, and add nitric acid until effervescence ceases and the liquid





remains distinctly acid. The liquid should be perfectly clear (absence of silica). Divide the liquid into three parts for the undermentioned tests.

*Chlorides:* To 10 mils of the solution add 1 mil silver nitrate solution; no opalescence should be immediately produced.

*Lead and Iron:* To 10 mils of the solution add sufficient ammonia solution to render the liquid faintly alkaline, then two drops ammonium sulphide solution; no darkening should occur.

*Calcium Salts:* Add ammonia to 10 mils of the solution in slight excess, and then 1 mil ammonium oxalate solution; no precipitate should immediately appear.

#### Trade Varieties.

Magnesium carbonate is sold in two varieties, known as heavy and light respectively. Their relative densities are as  $3\frac{1}{2}$  to 1, and commercial varieties usually contain traces of all the impurities mentioned above. Native magnesite, when finely ground and sifted, is sometimes sold as commercial carbonate.

## MAGNESIUM CHLORIDE.



White hygroscopic crystals.

#### Tests.

*Solubility:* Soluble in less than its own weight of water and about six times its weight of 90 per cent. alcohol.

*Sulphates:* Dissolve 1 Gm. in 20 mils of water, add 2 drops hydrochloric acid, and then 1 mil barium chloride solution; no turbidity should appear within five minutes.

*Iron and Heavy Metals:* Dissolve 1 Gm. in 20 mils of water, and add 5 drops of ammonium sulphide solution; no darkening should appear.

## MAGNESIUM OXIDE.



A white powder, nearly insoluble in water, but imparting to it a faint alkaline reaction. Soluble in acid, without effervescence.

#### Tests.

*Solubility:* The oxide should comply with the tests described under magnesium carbonate, using  $1\frac{1}{2}$  Gm. in place

of the 3 of carbonate, except that it should dissolve without effervescence in acids.

#### Trade Varieties.

The oxide, like the carbonate, is sold in two forms, heavy and light magnesia, which have about the same relative densities as the two carbonates. Burnt magnesite, obtained by calcining native magnesite, is frequently used for technical purposes, and can be obtained containing 90-95 MgO. Like magnesite, however, it leaves considerable quantities of silicious residue when treated with dilute acids, and contains considerable quantities of iron. It is frequently used in a large scale for neutralising acid liquids on account of its high neutralisation value, and the absence of effervescence during the process.

### MAGNESIUM OXIDE FREE FROM SULPHATES.

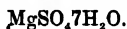
This resembles the magnesium oxide described above, but has been derived from nitrate or chloride of magnesium instead of sulphate, which is the usual source. When prepared from the last-mentioned salt it always contains traces of sulphate, which are carried down by the precipitated carbonate and not removed by washing.

#### Tests.

It should comply with the tests described above for magnesium oxide, and, in addition, should be tested for sulphates.

*Sulphates*: Suspend 1 Gm. in 50 mls of water, and add hydrochloric acid drop by drop, warming gently until the oxide is dissolved and a slightly acid liquid is obtained. Add 2 mls barium chloride solution, set aside for three hours; no precipitate should be produced.

### MAGNESIUM SULPHATE.



White, clear, transparent crystals, free from efflorescence, forming a neutral solution in water.

#### Tests.

*Solubility*: Soluble in  $1\frac{1}{2}$  times its weight of cold water; insoluble in alcohol.

*Chlorides*: Dissolve 1 Gm. in 20 mls distilled water, and add 1 mil silver nitrate solution; no turbidity should appear within two hours.







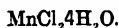
*Iron and Heavy Metals:* Dissolve 1 Gm. in 20 mls of water, add 5 drops ammonium sulphide solution; no darkening should occur.

#### Trade Varieties.

Under the name of Epsom Salts, this article is very largely used in medicine. The lower grades are commonly sold in sacks, and contain much foreign matter, and hence cannot give bright solutions in water. The purified article required by most pharmacopœias for use in medicine are fairly clean, and contain only small traces of iron.

The purified and recrystallised salt described above is in brighter and larger crystals than the ordinary Epsom Salts of trade.

## MANGANESE CHLORIDE.



Reddish-pink crystals. Hygroscopic.

#### Tests.

*Solubility:* Soluble in about its own weight of water.

*Sulphates:* 1 Gm. dissolved in 20 mls of water with 5 drops hydrochloric acid should give no precipitate on the addition of 1 mil barium chloride solution after standing one hour.

*Iron:* Dissolve 1 Gm. in 50 mls of water, add 5 drops hydrochloric acid and 1 mil chlorine water. Boil, cool and add 2 mls potassium thiocyanate solution: no red colour should be immediately produced.

*Magnesium and Alkalies:* Dissolve 2 Gms. in 50 mls of water, add 3 Gms. of ammonium carbonate dissolved in 50 mls of water, warm the mixture, set aside to cool and then filter. Evaporate the filtrate to dryness and ignite the residue, which should weigh not more than 1 Mgm.

*Calcium:* 1 Gm. dissolved in 50 mls of water with the addition of 2 mls ammonium oxalate solution should give no immediate precipitate.

*Zinc:* 1 Gm. dissolved in 20 mls of water with 5 drops acetic acid should give no precipitate on the addition of an equal bulk of hydrogen sulphide solution.

#### Trade Varieties.

Commercial manganese chloride is usually somewhat discoloured by various impurities, chiefly iron. Such samples also show traces of sulphuric acid.

## MANGANESE PEROXIDE.

(Manganese dioxide precipitated.)

Fine black powder. Permanent in the air, and consisting chiefly of the dioxide  $\text{MnO}_2$ .

### Tests.

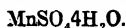
*Solubility:* Insoluble in water, dissolved by hot sulphuric acid with evolution of oxygen, and by hot hydrochloric acid with evolution of chlorine.

*Quantitative Determination:* Dissolve 0.2 Gm. in a mixture of 50 mls N/10 oxalic acid solution and 3 mls sulphuric acid in a flask heated on a water-bath. The resulting solution diluted with 100 mls warm water should require the addition of not more than 13 mls N/10 potassium permanganate solution to produce a permanent pink tint corresponding to over 80 per cent.  $\text{MnO}_2$ .

### Trade Varieties.

Recovered manganese in the form of a brownish-black powder is sold at a low price, and is suitable for the manufacture of chlorine. Native pyrolusite in the form of steel-grey lumps or in granules or powder of varying degrees of fineness is met with. The highest grades of pyrolusite should contain from 80 to 90 per cent.  $\text{MnO}_2$ , but sometimes samples containing only 25 per cent. are met with. Adulteration with carbonaceous matter has also been observed.

## MANGANESE SULPHATE.



Very pale rose-coloured crystals, or whitish-rose crystalline granular powder.

### Tests.

*Solubility:* Soluble in about its own weight of water. Insoluble in alcohol.

*Chlorides:* 1 Gm. dissolved in 40 mls of water with 1 ml nitric acid should give not more than the faintest opalescence on the addition of 2 mls silver nitrate solution.

*Iron, Magnesium, Alkalies, Calcium, and Zinc:* Test as described under manganese chloride.

### Trade Varieties.

Manganese sulphate is sold in commercial varieties which are often nearly pure, and almost free from iron. They contain variable proportions of water of crystallisation. Contamination with large proportions of alkalies is met with, and such impurities should be specially looked for. The sul-





phate sold for medicinal use is often in minute crystalline powder, which appears almost white; the large rose-coloured clear crystals are difficult to obtain.

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## MERCURY.

Liquid metal, which should present a lustrous surface, and no appearance of "tailing" when a drop is rolled about on the surface of a piece of white glazed paper. Heated in a porcelain dish it should volatilise entirely. Specific gravity, 13.573.

### Tests.

*Absence of Residue:* Heat 10 Gms. in a glazed porcelain dish in the fume chamber. It should volatilise, and leave no visible residue.

*Foreign Metals:* Boil 5 Gms. of mercury with 5 mils of water and 4.5 Gms. sodium thiosulphate for about one minute; the mercury should not lose its lustre, and should not acquire more than a slightly yellowish shade.

### Trade Varieties.

The mercury of commerce usually contains traces of other metals such as lead and iron, from which it is freed by careful distillation. Mechanical impurities can be separated by straining through a fine cloth or leather, and traces of foreign metals can often be removed by thorough agitation with potassium cyanide solution.

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## MERCURIC CHLORIDE.



White prismatic crystals dissolving in water forming a solution having a distinctly acid reaction.

### Tests.

*Solubility:* Soluble in about 20 parts of cold water. More freely soluble in alcohol and ether. 2 Gms. with 20 mils of ether should form a bright solution, and leave no insoluble residue.

*Fixed Residue:* 2 Gms. placed at the bottom of a dry test tube and carefully heated should volatilise and leave no visible residue.

*Foreign Salts:* Dissolve 1 Gm. in 25 mils of water, add 1 mil hydrochloric acid and completely saturate with hydrogen sulphide; allow it to stand two hours and filter. The filtrate should be colourless, and when evaporated on the water-bath should leave less than 2 Mgm. residue.

**Trade Varieties.**

Mercuric chloride occurs in commerce in translucent masses mixed with more or less powder, and usually leaves a slight residue when treated with solvents. Good varieties contain 99.5 per cent.  $\text{HgCl}_2$ .

**MERCURIC OXIDE.**

Dense orange-red or brick-red crystalline powder when produced by heating mercurous nitrate; or smooth dense orange-yellow powder when produced by precipitating a solution of mercuric salt with an alkaline hydroxide.

**Tests.**

*Solubility:* Insoluble in water, easily and entirely soluble in diluted hydrochloric or nitric acid forming colourless solutions.

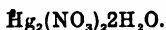
*Fixed Residue:* 1 Gm. heated to redness in a porcelain dish is decomposed into oxygen and mercury, and leaves a residue which should weigh less than 1 Mgm.

*Chlorides:* 0.1 Gm. dissolved in 10 mls of water and 1 mil nitric acid should form a clear solution giving only the faintest opalescence on the addition of 1 mil silver nitrate solution.

*Nitrates:* 1 Gm. mixed with 5 mls of water and 2 mls of sulphuric acid added, the mixture cooled and 2 mls ferrous sulphate solution carefully poured upon it should develop no brown zone at the line of contact on standing.

**Trade Varieties.**

The red oxide is met with in many shades varying from orange-red to brick-red, the latter shade being produced by grinding the crystals very fine. The yellow precipitated variety is very finely divided, and reacts on this account more quickly than the crystalline variety.

**MERCUROUS NITRATE.**

Colourless, transparent, tabular crystals.

**Tests.**

*Solubility:* Soluble in  $1\frac{1}{2}$  to 2 times their weight of cold water, the solution becoming turbid on the formation of oxynitrate when freely diluted.

*Fixed Residue:* 2 Gms. treated as described under mercuric chloride should leave no visible residue.







*Mercuric Salt*: 1 Gm. dissolved in 10 mils of water previously mixed with 0.25 mil nitric acid, and completely precipitated with the addition of hydrochloric acid should give a filtrate which when diluted with 50 mils of water and treated with hydrogen sulphide should show only a few black flocks of mercuric sulphide.

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## METAPHOSPHORIC ACID (GLACIAL).



Clear glassy lumps or sticks, deliquescent and easily soluble in water. It always contains some ammonium metaphosphate, as the free acid cannot be obtained in the fused glassy condition without this addition.

### Tests.

The acid should comply with the tests described for phosphoric acid, except that described for pyrophosphates and metaphosphates, taking for each test half the quantities therein prescribed. The acid should be dissolved in water and boiled for ten minutes in order to convert it into orthophosphoric acid.

### Trade Varieties.

Metaphosphoric acid is met with in grades of purity corresponding to those described under phosphoric acid, and usually contain soda in place of ammonia.

---

## MOLYBDIC ANHYDRIDE.



Nearly white powder, usually showing a very faint bluish tinge due to traces of the lower oxide of molybdenum. Should contain 100 per cent.  $\text{MoO}_3$  when determined as lead molybdate.

### Tests.

*Solubility*: Nearly insoluble in water, but easily soluble on the addition of excess of ammonia or potash.

*Ammonium Salts*: 1 Gm. dissolved in 20 mils of water with the addition of 1 Gm. of potassium or sodium hydroxide and the solution heated to boiling should evolve no ammonia.

*Phosphates*: Dissolve 5 Gms. in water and ammonia: the solution should respond to the test given under ammonium molybdate.

*Metals*: 1 Gm. dissolved in ammonia water should answer the test described under ammonium molybdate.

*Sulphates and Chlorides:* Shake 1 Gm. into 10 mils of water, set aside five minutes and filter; the filtrate should respond to the test described under ammonium molybdate.

#### Trade Varieties.

Commercial molybdic acid contains 80 to 85 per cent. of molybdic acid  $H_2MoO_4$  and usually heavy traces of alkalies.

## NITRIC ACID.



A colourless fuming liquid containing 70 per cent. of real acid. Boiling point,  $121^\circ$ . Specific gravity 1.42.

#### Tests.

*Residue:* 10 mils evaporated to dryness on the water-bath and the dish heated to  $120^\circ$  should leave less than 1 Mgm. residue.

*Sulphates:* 50 mils evaporated to 5 mils on a water-bath and the residue diluted with water to 50 mils, 5 mils barium chloride solution added and set aside twelve hours should remain perfectly clear.

Compare corresponding test under hydrochloric acid.

*Chlorides:* 5 mils diluted with water to 50 mils and 2 mils silver nitrate solution added should show no turbidity after two hours.

*Metals:* 5 mils diluted to 50 with distilled water and rendered alkaline by the addition of solution of ammonia should show no darkening on the addition of 1 mil ammonium sulphide solution.

#### Trade Varieties.

The acid having specific gravity 1.42 is the strength most frequently employed. An acid having a specific gravity 1.35 and containing 56 per cent. real acid is also frequently used. Fuming nitric acid specific gravity 1.5 contains about 95 per cent. real acid, and has a reddish colour due to the presence of oxides of nitrogen. This acid cannot be obtained so free from sulphates as the test given above requires. Commercial nitric acids always show more or less heavy traces of sulphates, and will not comply with the test for fixed residue given above.

## OXALIC ACID.



Colourless prismatic crystals. When heated in a water-bath it melts at  $98^\circ$  and becomes anhydrous on prolonged heating.





**Tests.**

*Solubility*: Soluble in 12 parts of water, less soluble in alcohol, sparingly in ether and insoluble in chloroform, benzene, and petroleum ether.

*Residue*: 2 Gms. heated over a small flame should leave no appreciable residue.

*Chlorides*: 2 Gms. dissolved in 20 mls of water with the addition of 2 mls nitric acid should give no turbidity on the addition of 1 mil silver nitrate solution.

*Sulphates*: 2 Gms. dissolved in 20 mls of water with the addition of 2 mls hydrochloric acid and 1 mil barium chloride solution should give no precipitate after setting aside for two hours.

*Metals*: 2 Gms. dissolved in 20 mls of water and the solution rendered alkaline by the addition of ammonia should show no darkening on the addition of 1 mil ammonium sulphide solution.

**Trade Varieties.**

Commercial oxalic acid is usually found in a more or less effloresced condition, and leaves an appreciable residue on ignition composed chiefly of alkali salts. The anhydrous acid is also met with in commerce, but usually contains some combined water unless recently prepared.

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**PERCHLORIC ACID.**

An aqueous solution containing 20 per cent.  $\text{HClO}_4$ . Specific gravity 1.120.

**Tests.**

*Residue*: 5 mls evaporated and gently ignited should yield a residue weighing less than 0.3 Mgm.

*Chlorides*: 1 mil diluted with 20 mls of water with 1 mil nitric acid should only give the faintest opalescence on the addition of 1 mil silver nitrate solution.

*Sulphates*: 1 mil diluted with 20 mls of water, and 1 mil hydrochloric acid should give no immediate turbidity on the addition of 1 mil barium chloride solution.

*Metals*: 2 mls diluted with 20 mls of water, made faintly alkaline with solution of ammonia and 0.5 mil ammonium sulphide added; the liquid should show no darkening.

*Barium*: 2 mls diluted with 20 mls of water and 0.5 mil dilute sulphuric acid should give no immediate turbidity.

**Trade Varieties.**

A cruder form than the above, made by the action of sulphuric acid on barium perchlorate, is obtainable in commerce, and contains fairly large traces of the impurities mentioned above.

**PHOSPHORIC ACID.**

A solution containing 66·3 per cent.  $\text{H}_3\text{PO}_4$  equivalent to 48 per cent.  $\text{P}_2\text{O}_5$ . Specific gravity 1·5. A colourless syrupy liquid miscible with water and alcohol.

**Tests.**

*Calcium*: 1 mil diluted with 20 mils of water and rendered distinctly alkaline by the addition of solution of ammonia should give no turbidity on the addition of 1 mil ammonium oxalate solution.

*Metals*: 1 mil diluted with 20 mils of water and rendered distinctly alkaline by the addition of solution of ammonia should show no darkening on the addition of 0·5 mil ammonium sulphide solution.

*Chlorides*: 1 mil diluted with 10 mils of water and 1 mil silver nitrate solution added should give no precipitate.

*Phosphites*: 1 mil diluted with 10 mils of water and 1 mil mercuric chloride solution added and the whole gently warmed: no turbidity should result.

*Metaphosphates and Pyrophosphates*: 2 mils diluted with 10 mils of water should give no precipitate on the addition of 5 mils ferric chloride solution, and setting aside one hour.

*Silica*: 2 mils diluted with 40 mils of water and set aside twelve hours should show no gelatinous deposit.

*Nitrates*: 2 mils mixed with 2 mils sulphuric acid, the mixture cooled, and a crystal of ferrous sulphate added: a brownish-black colour should appear around the crystal.

*Arsenic*: 2 mils diluted with 20 mils of water and introduced in the Marsh-Berzelius apparatus should show no arsenic stain after the action is continued thirty minutes.

**Trade Varieties.**

Phosphoric acid for medicinal purposes is sold in fairly pure condition, and in addition to the specific gravity described above is met with more concentrated, having specific gravity 1·75 and containing 90 per cent. phosphoric acid, and also in a more dilute form having a specific gravity 1·08 and containing 13·8 per cent. phosphoric acid. For technical use acids of lower degrees of purity than those described above are met with; these are often not colourless, and usually contain considerable quantities of calcium, iron and silica, as they are made from bone ash and not from phosphorus.







**PHOSPHORIC ANHYDRIDE.**

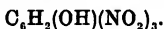
Snow-white flocculent powder, which combines with water with great violence. It must be stored in well-closed vessels, as it absorbs moisture very rapidly from the air, forming metaphosphoric acid.

**Tests.**

*Solubility*: 1 Gm. added cautiously in small portions to 20 mls distilled water should dissolve, forming a clear solution.

*Residue*: 1 Gm. heated in a dry tube should volatilise, leaving a residue weighing less than 0.01 Gm. This residue should be glassy and colourless.

*Arsenic*: The clear solution from the solubility test when mixed with equal volume of hydrogen sulphide solution should not show any yellow colouration or turbidity.

**PICRIC ACID.**

Pale yellow lustrous scales freely soluble in alcohol and ether, soluble in rather less than 50 parts of water. Melting point  $122.5^\circ$ .

**Tests.**

*Solubility*: Place 1 Gm. in a flask with 100 mls of water at  $50-60^\circ \text{C}$ . The acid should dissolve completely, and on setting the flask aside for half an hour no deposit of insoluble matter should be visible.

*Ash*: 1 Gm. cautiously heated in a shallow platinum basin should leave an ash weighing less than 2.0 Mgms.

*Sulphates*: Shake 2 Gms. with 20 mls distilled water at intervals during five minutes. Filter, and to the filtrate add 1 mil nitric acid and 1 mil barium chloride solution; no turbidity should appear.

**Trade Varieties.**

Picric acid of commerce is usually of fair purity, and melts at  $120-121^\circ \text{C}$ . It usually shows more ash and insoluble matter than in the tests above, and is frequently of a very pale shade and not well crystallised.

**PHOSPHORUS.**

Yellowish semi-transparent sticks about  $\frac{1}{4}$  in. in diameter, free from reddish incrustation. Melting point  $43.5^\circ \text{C}$ . Specific gravity 1.8.

**Tests.**

*Solubility*: Insoluble in water, slightly soluble in alcohol, more soluble in ether and chloroform, freely soluble in carbon bisulphide.

*Residue*: 1 Gm. boiled with 10 mls nitric acid and 10 mls of water should dissolve slowly and leave no residue.

*Sulphates*: Take half the solution in the preceding test, evaporate until no more nitrous fumes are evolved, dilute to 100 mls and add 2 mls barium chloride solution; only the faintest opalescence should be produced after standing five minutes.

#### Trade Varieties.

For commercial purposes phosphorus is sold in thick sticks and in slabs, which frequently contain particles of foreign matter but otherwise are of fair purity.

## PLATINUM PERCHLORIDE.



Reddish-brown crystalline masses, very deliquescent.

#### Tests.

*Solubility*: A 5 per cent. solution in water should be perfectly clear and pale yellow.

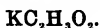
*Iridium*: The presence of this impurity is shown by the aqueous solution having a brownish tinge.

*Residue*: 1 Gm. carefully ignited in a porcelain crucible should leave a residue of spongy platinum weighing not less than 0.37 Gm. This residue when extracted with water should yield less than 2 Mgm. soluble solid residue.

#### Trade Varieties.

These may be incompletely soluble in water or alcohol, and when prepared from platinum scrap frequently form a brown solution, due to the presence of traces of iridium.

## POTASSIUM ACETATE.



White granular crystalline powder, or masses; very deliquescent. Melts a little below 300° C.

#### Tests.

*Solubility*: Soluble in about half its weight of water and 2 parts of alcohol. The aqueous solution is alkaline to litmus.

*Sulphates*: 1 Gm. with 20 mls of water and 2 mls hydrochloric acid should show no turbidity on the addition of 1 ml barium chloride solution after setting aside half an hour.

*Chlorides*: 1 Gm. with 30 mls of water and 2 mls nitric acid should show no immediate turbidity on the addition of 1 ml silver nitrate solution, and only the faintest opalescence on standing half an hour.





*Heavy Metals*: 2 Gms. with 20 mils of water and an equal volume of hydrogen sulphide solution should show no darkening after standing half-an-hour.

#### Trade Varieties.

The commercial salt occurs in lustrous masses, which are sometimes not colourless, and contain some sulphate and chloride derived from the potash salt used in manufacture.

## POTASSIUM BICARBONATE.



Colourless transparent prisms or powder.

#### Tests.

*Solubility*: Should dissolve in four times its weight of cold water. Insoluble in alcohol.

• *Chlorides*: 2 Gms. in 40 mils of water with 5 mils nitric acid warmed until effervescence ceases, and 1 mil silver nitrate solution added; only the faintest opalescence should be produced on standing two hours.

*Sulphates*: 2 Gms. with 40 mils of water and 5 mils hydrochloric acid and 1 mil barium chloride solution added; no precipitate should be produced after standing two hours.

*Nitrates*: 2 Gms. with 20 mils of water and 2 mils sulphuric acid heated until effervescence ceases and a crystal of ferrous sulphate dissolved in the cool liquid; on pouring sulphuric acid down the side of the tube no brown zone should be produced at the junction of the two liquids.

*Iron and Heavy Metals*: 4 Gms. dissolved in 20 mils of water and an equal volume of hydrogen sulphide added should show no darkening colour.

#### Trade Varieties.

Commercial potassium bicarbonate is usually of very fair purity, containing only traces of the impurities mentioned above, and sometimes leaving a slight residue when dissolved in water.

## POTASSIUM BICHROMATE.



• Red crystals melting when heated to a brown liquid, which when further heated leaves a residue of potassium chromate and chromium trioxide.

#### Tests.

*Solubility*: Soluble in 10 parts of water, forming an acid solution; insoluble in alcohol.

*Chlorides*: 1 Gm. in 50 mils of water with 5 mils nitric acid and 1 mil of silver nitrate solution added, should show no immediate turbidity. •

*Sulphates*: 1 Gm. with 50 mils of water and 5 mils hydrochloric acid and 1 mil barium chloride solution should show no precipitate on standing two hours.

*Alkaline Earths*: 1 Gm. dissolved in 20 mils of water and made distinctly alkaline with solution of ammonia should show no turbidity on adding 1 mil ammonium oxalate solution and setting aside two hours.

#### Trade Varieties.

The commercial salt in large crystalline masses contains 97-98 per cent.  $K_2Cr_2O_7$ . It is frequently incompletely soluble in water, and usually contains considerable traces of sulphate.

## POTASSIUM BISULPHATE.



Colourless crystals or fragments of fused cakes.

#### Tests.

*Solubility*: Forms a clear solution with twice its weight of water.

*Chlorides*: 1 Gm. in 20 mils of water gives no turbidity on the addition of 1 mil silver nitrate solution and setting aside for two hours.

*Heavy Metals*: 1 Gm. in 20 mils of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron*: 1 Gm. in 20 mils of water made alkaline with solution of ammonia should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Arsenic*: 1 Gm. dissolved in 10 mils of water and introduced into the Marsh-Berzelius apparatus should show no arsenical stain after thirty minutes' action.

## POTASSIUM BITARTRATE.



Dry white crystalline powder, which shows no loss in weight on drying at  $100^\circ$  C. for one hour.

#### Tests.

*Solubility*: Soluble in about 200 parts of water, giving an acid solution. Insoluble in alcohol.

*Chlorides*: 1 Gm. with 20 mils of water and 1 mil of nitric acid should show only the faintest opalescence on adding 1 mil silver nitrate solution and setting aside thirty minutes.

*Sulphates*: 1 Gm. with 20 mils of water and 2 mils of hydrochloric acid should show no turbidity after adding 1 mil barium chloride solution and setting aside two hours.







*Heavy Metals*: 2 Gms. with 20 mils of water and 10 mils of ammonia solution should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

#### Trade Varieties.

Commercial potassium bitartrate, known as cream of tartar, when dried, should contain at least 97·5 per cent.  $\text{KHC}_4\text{H}_4\text{O}_6$ .

## POTASSIUM BROMIDE.

$\text{KBr}$ .

Colourless cubical crystals.

#### Tests.

*Solubility*: Soluble in 1·7 parts of water, and very slightly soluble in alcohol.

*Free Alkali*: A 5 per cent. aqueous solution of the salt should not affect neutral litmus paper.

*Heavy Metals and Iron*: 1 Gm. in 20 mils of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution, even after the addition of 1 mil of solution of ammonia.

*Sulphates*: 1 Gm. in 20 mils of water should show no turbidity after adding 1 mil of barium chloride solution and setting aside for two hours.

*Thiocyanates*: 1 Gm. in 20 mils of water should give a yellow and not a red or reddish-brown colouration on the addition of two drops of ferric chloride solution.

*Chloride*: Freshly powdered and dried 1 Gm. of the pure salt titrated with N/10  $\text{AgNO}_3$  solution will require 83·95 C.c.; it should not require more than 84·5, corresponding to about 1 per cent. potassium chloride.

#### Trade Varieties.

The potassium bromide used in medicine is usually of fair purity, but samples of American origin have been found to contain 5 to 6 per cent. of chloride. The other impurities likely to be met with are moisture, free alkali, and traces of iron.

## POTASSIUM CARBONATE.

$\text{K}_2\text{CO}_3$ .

A white, deliquescent, finely granular powder.

#### Tests.

*Solubility*: Soluble in about its own weight of water; insoluble in alcohol. With 5 parts of water it should form a perfectly clear solution.

*Chlorides*: 1 Gm. with 20 mils of water, and sufficient nitric acid to acidify the solution, should give only the faintest opalescence with 1 mil silver nitrate solution.

**Sulphates:** 1 Gm. with 20 mils of water, with sufficient hydrochloric acid to acidify the solution, should give no turbidity after adding 1 mil barium chloride solution and setting aside two hours.

**Heavy Metals and Iron:** 1 Gm. with 20 mils of water should give no darkening on the addition of an equal volume of hydrogen sulphide solution.

**Nitrate:** Dissolve 0.5 Gm. in 5 mils of water, add dilute sulphuric acid until the liquid has a faintly acid reaction, and dissolve in it a crystal of ferrous sulphate. Then pour down the side of the tube sufficient strong sulphuric acid to form a distinct layer; no brown zone should appear at the junction of the two liquids.

**Quantitative Determination:** Owing to its hygroscopic nature the salt usually contains 1 to 2 per cent. of water. When carefully stored it should show not less than 98 per cent.  $K_2CO_3$  by titration with normal acid, 1 C.c. of which = 0.069 Gm.  $K_2CO_3$ .

#### Trade Varieties.

Potassium carbonate is used for many purposes in the arts, and is consequently met with in very varying degrees of purity.

Salt of tartar contains about 82 per cent.  $K_2CO_3$  and 16 per cent. water. American potash is a very crude form of hydroxide containing carbonate, having a grey or green colour.

Pearlash is in white granular masses, and may contain 10-20 per cent. sulphate.

## POTASSIUM CHLORATE.



Colourless tabular crystals.

#### Tests.

**Solubility:** 1 Gm. gently warmed with 16 mils of water forms a clear neutral solution. Insoluble in absolute alcohol.

**Alkaline Earths:** 1 Gm. dissolved in 20 mils of water and 1 mil ammonium oxalate solution added should show no turbidity after standing two hours.

**Chlorides:** 1 Gm. dissolved in 20 mils of water and 1 mil of silver nitrate solution added should show no turbidity after standing two hours.

**Sulphates:** 1 Gm. dissolved in 20 mils of water and 1 mil barium chloride added should show no turbidity after standing two hours.

**Heavy Metals:** 2 Gms. dissolved in 50 mils of water and an equal volume of hydrogen sulphide solution added should show no darkening.

**Arsenic:** Reduce 10 Gms. of the salt to coarse powder, place in a porcelain dish, gently warm, and add, in small portions,





40 mils of hydrochloric acid. Evaporate to dryness on a water-bath, dissolve the residue in 30 mils of water, and place this solution in the Marsh-Berzelius apparatus. No arsenical stain should appear in the tube after thirty minutes' action.

#### Trade Varieties.

Commercial potassium chlorate is usually of good purity. It can be purchased in several forms—large tabular crystals, santonin-crystals, and in powder. The commercial powder sometimes contains mechanical impurities, and gives an imperfect solution. The purest form is usually in small tabular crystals obtained by recrystallisation from moderately concentrated solutions.

## POTASSIUM CHLORIDE.



Colourless translucent cubical crystals.

#### Tests.

*Solubility:* Forms a clear neutral solution in 4 parts of water. Insoluble in absolute alcohol.

*Sulphates:* 1 Gm. dissolved in 20 mils of water and 1 mil of barium chloride solution added should show no turbidity after standing two hours.

*Alkaline Earths:* 1 Gm. dissolved in 20 mils of water and 1 mil ammonium oxalate solution added should show no turbidity after standing two hours.

*Iron:* 1 Gm. dissolved in 20 mils of water with 1 mil each of nitric acid and potassium thiocyanate solution should show no red tint even after gently warming.

*Heavy Metals:* 2 Gms. dissolved in 50 mils of water and an equal volume of hydrogen sulphide solution added should show no darkening.

#### Trade Varieties.

Potassium chloride is sold for agricultural purposes in many grades of purity from 60 per cent. KCl upwards. The lower grades are usually dark and dirty. The commercially pure form in white crystals usually contain only small quantities of sulphates and metals.

## POTASSIUM CHROMATE.



Yellow translucent crystals.

#### Tests.

*Solubility:* Forms a clear solution with 3 parts of water. Insoluble in alcohol.

*Sulphates*: 1 Gm. in 20 mls of water with 5 mls of hydrochloric acid should give no turbidity with 1 mil barium chloride solution after setting aside two hours.

*Chlorides*: 1 Gm. in 50 mls of water with 4 mls nitric acid gently warmed should show no immediate turbidity on the addition of 2 mls silver nitrate solution.

*Alkaline Earths*: 1 Gm. in 20 mls of water with 1 mil each of ammonia and ammonium oxalate solutions should show no turbidity after setting aside two hours.

#### Trade Varieties.

Commercial potassium chromate in good crystals is usually of fair purity, but not sufficiently good for use as an indicator in volumetric work.

## POTASSIUM CYANIDE.

### KCN.

Colourless granular crystalline powder smelling of hydrocyanic acid.

#### Tests.

*Solubility*: Forms with 3 parts of water a clear colourless solution having an alkaline reaction. Very slightly soluble in absolute alcohol, but dissolving more freely in weaker alcohol.

*Carbonate*: 1 Gm. dissolved in 20 mls of water and 3 mls of hydrochloric acid should show no effervescence, or at most only a few bubbles of gas should be disengaged.

*Sulphate*: The liquid from the preceding test should show no turbidity on adding 1 mil barium chloride solution and setting aside thirty minutes.

*Titration*: When treated with N/10 silver nitrate the salt should show at least 96 per cent. KCN. When freshly crystallised and carefully dried it may be obtained 98 to 99 per cent. It should be carefully stored in a dry and cool place.

#### Trade Varieties.

Commercial potassium cyanide occurs in fused cakes or masses, and was formerly obtained by fusing potassium ferrocyanide with potassium carbonate. This variety always contained cyanate. The chief varieties now in commerce are:—

(a) 98 per cent. "double" salt containing a mixture of potassium and sodium cyanides equivalent to 98 per cent. KCN when titrated with silver nitrate.

(b) 95 per cent. "single" salt, containing 95 per cent. KCN when titrated with silver nitrate.

(c) "Gold" cyanide containing 70 per cent. KCN.

(d) "Silver" cyanide containing 40 per cent. KCN.







The varieties (c) and (d) are used for making, respectively, gold and silver solutions for electro-plating, and are being displaced by the higher-strength cyanides now available.

For gold extraction sodium cyanide equivalent to 128 to 130 per cent. KCN is largely used in place of potassium cyanide on account of its greater value in cyanogen.

## POTASSIUM FERRICYANIDE.



Ruby-red crystals, with a shining surface, free from powdery incrustation.

### Tests.

*Solubility*: 1 Gm. dissolves completely to a clear red solution in 3 mls of water.

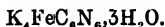
*Ferrocyanide*: Take a crystal weighing about 0.25 Gm., shake it rapidly with 20 mls of water to wash the surface; reject the liquor and dissolve the remaining crystal in 50 mls of water; on adding 1 drop of ferric chloride solution no blue colour should appear.

*Sulphate*: 1 Gm. in 20 mls of water and 1 mil hydrochloric should give no turbidity on the addition of 1 mil barium chloride solution. If the tube be left undisturbed for six hours and the liquid carefully decanted no deposit of barium sulphate should be visible in the bottom of the tube.

### Trade Varieties.

Commercial potassium ferricyanide is usually incompletely soluble in water.

## POTASSIUM FERROCYANIDE.



Soft lemon-yellow tabular or prismatic crystals, slightly efflorescent.

### Tests.

*Solubility*: 1 Gm. dissolves in 3 mls of water to a clear pale yellow neutral solution.

*Sulphate*: 0.5 Gm. in 20 mls of water and 1 mil hydrochloric acid should give no turbidity on adding 1 mil barium chloride solution and setting aside thirty minutes.

### Trade Varieties.

Commercial samples are usually of fair purity, containing traces of chlorides and sulphates, but do not make a clear solution in water.

**POTASSIUM HYDROXIDE.****KOH.**

White lumps, very deliquescent, and showing a crystalline fracture. It is not desirable to employ this variety in the form of sticks on account of the unavoidable exposure to air in casting in moulds and the risk of metallic contamination.

**Tests.**

*Solubility:* 5 Gms. dissolve to a clear colourless solution in 10 mils of water. Dilute the resulting solution to 100 mils for employment in the following tests:—

*Carbonate:* To 10 mils of the above solution add 5 mils dilute hydrochloric acid; no bubbles of gas should be visible.

*Sulphates:* To 10 mils of the solution add 2 mils of hydrochloric acid and 1 mil barium chloride solution; no turbidity should appear even after standing six hours.

*Chlorides:* To 10 mils of the solution add 3 mils of nitric acid and 1 mil silver nitrate solution; only the faintest opalescence should appear.

*Nitrates:* To 10 mils of the solution add an equal volume of sulphuric acid and then 5 drops of a saturated solution of metaphenylene diamine; no blue colour should appear.

*Silica:* To 10 mils of the solution add 3 mils of hydrochloric acid and evaporate on a water-bath to dryness. Heat for a further thirty minutes and then dissolve the residue in 20 mils of water, with the addition of 2 mils of hydrochloric acid. The resulting solution should be clear and free from any flocculent, insoluble matter.

*Alumina:* To 10 mils of the solution add 5 mils acetic acid, testing the resulting fluid with blue litmus paper to ensure its being acid; then add solution of ammonia, drop by drop, until the liquid is distinctly alkaline. Warm gently for half-an-hour, then set aside for another half-an-hour. No gelatinous flocks of aluminium hydrate should be visible.

*Iron:* The clear liquid from the alumina test should show no darkening on the addition of five drops ammonium sulphide solution previously diluted with 5 mils of water.

**Trade Varieties.**

Crude caustic potash is sold in various grades of purity for technical use. These are usually described in terms of percentage of  $K_2O$ . The cruder forms are grey or coloured by various impurities. The commercial potash sold in sticks for laboratory use should be white and not efflorescent from the presence of carbonate. It contains always considerable proportions of chlorides and sulphates, as well as the other impurities mentioned above, and contains about 80 per cent. KOH.





A better variety, described as "purified by alcohol," contains traces of chloride, but no sulphate, and only small traces of the other common impurities. It is impossible to prepare 100 per cent. KOH, and the highest percentage met with in commerce is about 85 per cent.

## POTASSIUM IODIDE.

### KI.

White cubical crystals becoming coloured when exposed to air, particularly if containing no free alkali.

#### Tests.

*Solubility*: Forms a perfectly clear solution with its own weight of water.

*Free Alkali*: 1 Gm. in 20 mls of freshly boiled and cooled water should show no red colour on the addition of one drop of phenol-phthalein solution.

*Iodate*: 1 Gm. in 20 mls of water with 2 mls each of starch solution and tartaric acid solution should not immediately become blue.

*Sulphate*: 1 Gm. in 20 mls of water with 2 mls dilute hydrochloric acid should show no turbidity after the addition of 1 mil barium chloride solution and setting aside one hour.

*Metals*: 1 Gm. in 20 mls water should show no darkening on the addition of an equal volume of hydrogen sulphide solution and 1 mil ammonia solution.

*Chlorides and Bromides*: Dissolve 0.2 Gm. in 6 mls dilute ammonia solution, add 13 mls N/10 silver nitrate solution, shake thoroughly, and filter. To the filtrate add sufficient nitric acid to give a distinct acid reaction; only the faintest turbidity should immediately appear.

#### Trade Varieties.

Potassium iodide is used in large quantities for medicinal purposes and usually contains a trace of free alkali, which renders it less liable to become brown from presence of free iodine in keeping. Traces of chloride and sulphate are often present in this variety. Iodate is not met with so often as formerly, probably because the salt is not usually made by the action of iodine on potassium hydroxide.

## POTASSIUM IODATE.

### KIO<sub>3</sub>.

White granular crystalline powder.

#### Tests.

*Solubility*: 1 Gm. forms a clear solution in 20 mls of water.

*Iodide*: 1 Gm. in 20 mls of water with 2 mls each of starch solution and tartaric acid solution should not immediately become blue.

**POTASSIUM NITRATE.**

Colourless, transparent prismatic crystals or white crystalline powder.

**Tests.**

*Solubility*: 2 Gms. forms with 10 mils of water a clear solution, having a neutral reaction.

*Sulphate*: 1 Gm. in 20 mils of water, with 1 mil of hydrochloric acid should show no turbidity on the addition of 1 mil barium chloride solution and setting aside two hours.

*Chloride*: 1 mil with 20 mils of water and 1 mil nitric acid should show no opalescence on adding 1 mil silver nitrate solution and setting aside two hours.

*Metals*: 1 Gm. in 20 mils of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron*: 1 Gm. in 20 mils of water with 5 mils nitric acid should show no red colour on the addition of 1 mil potassium thiocyanate solution.

**Trade Varieties.**

The saltpetre of commerce is usually of fair purity, chlorides and sulphate being usually present.

**POTASSIUM OXALATE.**

Colourless crystals forming a neutral solution in water.

**Tests.**

*Solubility*: 1 Gm. forms a clear solution with 5 mils of water.

*Chlorides*: 1 Gm. with 20 mils of water and 1 mil nitric acid shows no opalescence on the addition of 1 mil silver nitrate solution.

*Sulphates*: 1 Gm. in 20 mils of water with 1 mil hydrochloric acid should show no turbidity on adding 1 mil barium chloride solution and setting aside two hours.

*Metals*: 1 Gm. in 20 mils of water with 1 mil ammonia solution should show no darkening on the addition of an equal volume of hydrogen sulphide.

**Trade Varieties.**

Commercial potassium oxalate contains chlorides and sulphates and usually traces of iron and other metals.







**POTASSIUM PERMANGANATE.**

Purple prismatic crystals having a metallic lustre and free from dust or powder.

**Tests.**

*Solubility:* Shake 1 Gm. with 50 mls of water until the crystals have dissolved, and then set aside for two hours. Carefully decant the deep purple solution; the lower portion of the liquid should be transparent and free from any slimy deposit.

*Chloride, Sulphate, and Nitrate:* Dissolve 1 Gm. in 50 mls of water, add 10 mls of alcohol, heat nearly to boiling and maintain at that temperature until the permanganate is entirely reduced and the supernatant liquor becomes colourless. Filter and to 10 mls of the filtrate add 0.5 mil nitric acid and 1 mil silver nitrate solution; to another 10 mls add 1 mil hydrochloric acid and 1 mil barium chloride solution; no turbidity should immediately appear in either case, and even on standing thirty minutes only the faintest opalescence. To another 5 mls of the filtrate add 1 drop of diphenylamine solution, and then 1 mil sulphuric acid poured down the side of the tube; no blue colour should appear at the line of contact of the two fluids.

**POTASSIUM SULPHATE.**

White, dry, translucent crystals.

**Tests.**

*Solubility:* Should form a clear neutral solution with ten times its weight of water. Insoluble in alcohol.

*Chlorides:* 1 Gm. in 20 mls of water should show no turbidity on the addition of 1 mil silver nitrate solution.

*Heavy Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equivalent volume of hydrogen sulphide solution.

*Iron:* 1 Gm. in 20 mls of water with 3 drops nitric acid should not show a pink tint on the addition of 1 mil potassium sulphocyanide solution.

**Trade Varieties.**

Potassium sulphate of many varying grades of purity is met with on the market. The lower grades are greyish and are incompletely soluble in water. The ordinary recrystallised salt of commerce is usually fairly pure.

**POTASSIUM THIOCYANATE.****KCNS.**

Colourless, transparent, deliquescent crystals.

**Tests.**

*Solubility*: Easily soluble in water, forming a clear, colourless solution. Should also be completely soluble in 10 parts of alcohol.

*Sulphates*: 1 Gm. in 20 mls distilled water and 3 drops hydrochloric acid should show no immediate turbidity on the addition of 1 mil barium chloride solution.

*Heavy Metals*: 1 Gm. in 20 mls of water should show no darkening on the addition of an equivalent volume of hydrogen sulphide solution.

**Trade Varieties.**

Commercial samples are usually not quite colourless, and often show a pink tint due to the presence of iron. The salt should be kept in a cool place in well-stoppered bottles.

**SILVER NITRATE.****AgNO<sub>3</sub>.**

Colourless transparent tabular crystals darkening on exposure to air.

**Tests.**

*Solubility*: Soluble in less than its own weight of water and in about 20 parts of alcohol. The solutions have an acid reaction to litmus.

*General Impurities*: Dissolve 1 Gm. in 30 mls of water and warm the solution and add 1 mil hydrochloric acid. Heat to boiling, filter, and evaporate the filtrate to dryness; there should be no residue, or one weighing not more than 0.5 mg.

**Trade Varieties.**

The silver nitrate of commerce is usually very pure, but sometimes contains a trace of alkali salts and copper.

**SODA LIME.**

White porous granules of various sizes consisting of an intimate mixture of calcium and sodium hydroxides.

**Tests.**

*Solubility*: 1 Gm. suspended in 20 mls of water and hydrochloric acid added, drop by drop, until the latter is slightly in excess should yield a clear solution.





*Carbonate*: During the solution described in the previous test only a few bubbles of gas should escape.

*Nitrogen*: 1 Gm. placed in the bottom of a clear glass tube and damped with a few drops of water, and afterwards heated to a low redness, should evolve no fumes having an alkaline reaction to red litmus paper.

#### Trade Varieties.

Soda lime made from ordinary slaked lime and commercial caustic soda usually has a greyish tint, but is sufficiently good for the absorption of moisture in desiccators, etc.

## SODIUM.

Na.

A silvery white metal which can be easily cut with a knife. The freshly-cut surface has a metallic lustre, which quickly becomes dull on exposure to the air. It is usually preserved in mineral naphtha.

#### Tests.

*General Impurities*: Take 1 Gm. of the metal and carefully remove the adherent naphtha with clean blotting paper, cut it in small pieces, and add this a piece at a time to 20 mls of cold water contained in a basin covered with a glass plate. When the reaction is complete the solution should be colourless and clear. When warmed it should have no odour of ammonia, and should show no darkening on the addition of an equivalent volume of hydrogen sulphide solution.

#### Trade Varieties.

For commercial purposes sodium is supplied in large rectangular blocks, which are packed and preserved in sealed tins. The metal is usually pure, but not unfrequently masses of salt are found to be imbedded in the interior of the blocks.

## SODIUM ACETATE



Colourless, transparent, prismatic crystals, efflorescing in dry air.

#### Tests.

*Solubility*: Soluble in its own weight of water, forming a clear solution, which is slightly alkaline to litmus paper.

*Sulphates*: 1 Gm. in 20 mls of water should show no turbidity on the addition of 1 ml barium chloride solution and setting aside one hour.

*Chlorides*: 1 Gm. in 20 mls of water should show no turbidity on the addition of 1 mil silver nitrate solution and setting aside half an hour.

*Metals*: 1 Gm. in 20 mls of water should show no darkening on the addition of an equal bulk of hydrogen sulphide solution.

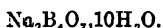
#### Trade Varieties.

Sodium acetate, if in clean white crystals, is usually fairly pure, but nearly always contains small quantities of iron.

## SODIUM AMALGAM.

Grey fragments, which can be broken with a hammer, and show a metallic lustre on the freshly broken surface. It is made containing from 2 to 8 per cent. of metallic sodium. The determination of the sodium content is carried out by adding a suitable quantity to water, avoiding loss of the solution during the action, and titrating the resulting solution of sodium hydroxide with standard acid.

## SODIUM BIBORATE.



Borax. Colourless, transparent prisms, slightly efflorescent in dry air.

#### Tests.

*Solubility*: Soluble in about 25 parts of water, forming a solution alkaline to litmus. More soluble in glycerin and insoluble in alcohol.

*Chlorides*: 0.5 Gm. in 20 mls of water, with 1 mil nitric acid added, should show no turbidity on the addition of 1 mil silver nitrate solution.

*Sulphate*: 0.5 Gm. in 20 mls of water, with 1 mil nitric acid, should show no turbidity on the addition of 1 mil barium chloride solution.

*Iron and Heavy Metals*: 0.5 Gm in 20 mls of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

#### Trade Varieties.

Commercial borax is usually nearly pure, unless intentionally adulterated. Another variety of borax containing five molecules of water can be obtained. The crystals are octahedral, and are prepared by crystallising hot solutions.

*Glass of Borax* is in the form of colourless, glassy, trans-







parent lumps, obtained by fusing borax until the whole of the water of crystallisation is removed.

*Calcined Borax.*—This is in the form of white, light, spongy masses, obtained by heating borax until most of the water of crystallisation is removed.

## SODIUM BICARBONATE.



White crystalline powder.

### Tests.

*Solubility:* Soluble in 12 parts of water, forming a clear solution and having an alkaline reaction to litmus paper.

*Chlorides:* 0.5 Gm. in 20 mls of water with 1 mil nitric acid should show no immediate opalescence on the addition of 1 mil silver nitrate solution.

*Sulphates:* 1 Gm. in 20 mls of water and 2 mls hydrochloric acid should give no turbidity on the addition of 1 mil barium chloride solution and setting aside two hours.

*Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equal quantity of hydrogen sulphide solution.

*Iron.*—1 Gm. in 2 mls of water and 1 mil nitric acid should show no pink tint on the addition of 1 mil potassium thiocyanate solution.

*Carbonate.*—1 Gm. dissolved quickly without agitation in 20 mls of water at a temperature below 15° gives a solution which is not reddened at once by the addition of 2 drops phenolphthalein solution.

*Ammonia.*—Heat 1 Gm. in the bottom of a dry test-tube, and suspend a piece of moistened red litmus paper in the upper part of the tube; the litmus paper should not be changed to blue.

*Silica.*—Dissolve 2.5 Gms. in a slight excess of dilute hydrochloric acid, evaporate to dryness in a platinum basin, heat the residue to between 120° and 150° C. for half an hour. To the cooled residue add 10 mls of water and 1 mil hydrochloric acid, decant the solution into a clean glass beaker, and examine carefully against the light; the solution should be quite free from suspended matter.

*Thiocyanate.*—1 Gm. with 20 mls of water with 1 mil nitric acid should show no red colour on the addition of 1 drop ferric chloride solution.

**Trade Varieties.**

Commercial bicarbonate of soda can now be purchased of very fair purity. It usually contains some carbonate with traces of chloride and sulphate, and occasionally one or other of the impurities mentioned above.

**SODIUM BISMUTHATE.**

A fine brown powder, insoluble in water, used in the determination of manganese in iron, steel, and alloys.

**Tests.**

*Oxidising Power:* Prepare a solution of hydrogen peroxide approximately decinormal as follows: Take 3.9 Gms. of sodium peroxide free from chlorides and add to 500 mls of water, then add 220 mls of a solution of nitric acid containing 315 Gms. real  $\text{HNO}_3$  per litre. Titrate this solution with  $\text{N}/10 \text{ KMnO}_4$  and adjust its strength to  $\text{N}/10 \text{ H}_2\text{O}_2$ . To 25 mls of this  $\text{N}/10 \text{ H}_2\text{O}_2$  solution add 0.25 Gm. of the sodium bismuthate in fine powder. When dissolved titrate the excess of  $\text{H}_2\text{O}_2$  with  $\text{N}/10 \text{ KMnO}_4$ , and thus determine the amount of  $\text{H}_2\text{O}_2$  decomposed by the bismuthate. 1 Gm. of the reagent should have an oxidising power equivalent to 58.61 mls  $\text{N}/10 \text{ KMnO}_4$ .

**SODIUM BISULPHATE FUSED.**

White cakes or fragments giving a strongly acid solution in water.

**Tests.**

*Chlorides.*—1 Gm. in 20 mls of water should show no turbidity within half an hour after the addition of 1 mil silver nitrate solution.

*Arsenic.*—1 Gm. in Marsh-Berzelius apparatus should show no arsenical stain after half-hour's treatment.

*Metals.*—1 Gm. in 20 mls of water should show no darkening on the addition of an equal volume hydrogen sulphide solution.

*Iron.*—1 Gm. in 20 mls of water rendered distinctly alkaline by the addition of ammonia should show no darkening on the addition of 1 mil ammonium sulphide solution.

**Trade Varieties.**

The crude form of sodium bisulphate known as "Nitric Cake," is obtained in the manufacture of nitric acid from sodium nitrate. The proportion of sulphuric acid is somewhat variable, and it sometimes contains undecomposed nitrate, and hence gives off nitric acid on further heating.





**SODIUM CARBONATE ANHYDROUS.**

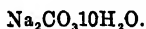
White powder, containing 98-100 per cent. of sodium carbonate.

**Tests.**

*Impurities:* This salt should be tested for impurities as described under "Sodium Bicarbonate," using half the quantity of salt therein prescribed.

**Trade Varieties.**

Soda crystals or washing soda is now in the market of high degree of purity, the total amount of impurities not being more than a few units per cent. Commercial anhydrous soda known as "Soda Ash" can be purchased containing 95 per cent. and upwards  $\text{Na}_2\text{CO}_3$ . It usually forms a turbid solution in water, and contains distinct traces of iron.

**SODIUM CARBONATE CRYST.**

Colourless transparent crystals readily efflorescing on exposure to air.

**Tests.**

*Solubility:* Soluble in 1.6 of water, insoluble in alcohol.

*Other Impurities:* This salt should be examined for impurities as described under "Sodium Bicarbonate," using, however, twice the amount of salt therein prescribed.

**SODIUM CHLORIDE.**

White cubical crystals forming a neutral solution.

**Tests.**

*Solubility:* Soluble in rather less than three parts of water, sparingly soluble in alcohol.

*Sulphates:* 1 Gm. in 20 mils of water with 1 mil hydrochloric acid should show no turbidity on adding 1 mil barium chloride solution and setting aside three hours.

*Alkaline Earths:* 1 Gm. in 30 mils of water should show no turbidity on the addition of 1 mil ammonium oxalate solution.

*Metals:* 1 mil in 20 mils of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron*: 1 Gm. in 20 mls of water should show no darkening on the addition of 1 mil ammonium sulphide solution.

*Magnesium*: 1 Gm. in 10 mls of water with the addition of 1 mil each ammonium hydroxide solution and ammonium phosphate solution should show no deposit after agitation and setting aside two hours.

#### Trade Varieties.

Table salt usually contains sufficient magnesium or calcium chloride to render the salt deliquescent, together with traces of iron. Rock salt and sea salt occur in the form of crystalline masses more or less coloured from the presence of iron. Pure sodium chloride is not hygroscopic.

## SODIUM CHLORIDE FUSED.



The above salt in the form of translucent crystalline masses obtained by fusing and cooling.

#### Tests.

It should answer the tests prescribed for the crystalline form.

## SODIUM HYDROXIDE.



White sticks or crystalline fragments, very deliquescent and caustic. The lump form is to be preferred to the stick on account of the exposure to air in casting and the risk of metallic contamination from the moulds.

#### Tests.

*Solubility*: Soluble in about its own weight of water and very soluble in alcohol. 5 Gms. should dissolve to a clear, colourless solution in 10 mls of water. Dilute the resulting solution to 100 mls for employment in the following tests.

*Carbonate*: To 10 mls of the above solution add 5 mls dilute hydrochloric acid; no bubbles of gas should be visible.

*Sulphates*: To 10 mls of the solution add 2 mls of hydrochloric acid and 1 mil barium chloride; no turbidity should appear even after standing six hours.

*Chlorides*: To 10 mls of the solution add 3 mls of nitric acid and 1 mil silver nitrate solution; only the faintest opalescence should appear.

*Nitrates*: To 10 mls of the solution add an equal volume of sulphuric acid and then 5 drops of a saturated solution of metaphenylene diamine: no blue colour should appear.







*Silica:* To 10 mls of the solution add 3 mls hydrochloric acid and evaporate on a water-bath to dryness. Heat for a further thirty minutes and then dissolve the residue in 20 mls of water with the addition of 2 mls hydrochloric acid; the resulting solution should be clear and free from any flocculent insoluble matter.

*Alumina:* To 10 mls of the solution add 5 mls acetic acid, testing the resulting fluid with blue litmus paper to ensure its being acid, then add solution of ammonia drop by drop until the liquid is distinctly alkaline. Warm gently for half an hour, then set aside for another half an hour; no gelatinous flocks of aluminium hydrate should be visible.

*Iron:* The clear liquid from the alumina test should show no darkening on the addition of five drops ammonium sulphide solution previously diluted with 5 mls of water.

#### Trade Varieties.

Crude caustic soda in the form of lumps can be purchased containing varying percentages of caustic soda, which are generally expressed in terms of  $\text{Na}_2\text{O}$ . A very convenient form is that known as "Granulated," consisting of the dried substance ground into coarse powder, and containing 98 per cent.  $\text{NaOH}$ . The ordinary stick soda of commerce is fairly clean, and contains 85-90 per cent.  $\text{NaOH}$ , with considerable quantities of the impurities mentioned above. Another variety known as purified by alcohol usually approaches in purity the standard given above. Caustic soda made by the action of metallic sodium and water is very pure when carefully prepared, and contains usually over 95 per cent.  $\text{NaOH}$ .

## SODIUM NITRATE.



Colourless transparent crystals.

#### Tests.

*Solubility:* Soluble in less than two parts of water; very sparingly soluble in alcohol.

*Sulphate:* 1 Gm. in 20 mls of water with 1 mil hydrochloric acid should show no turbidity on the addition of 1 mil barium chloride solution and setting aside two hours.

*Chlorides:* 1 Gm. with 20 mls of water and 1 mil nitric acid should show no opalescence on adding 1 mil silver nitrate solution and setting aside two hours.

*Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron:* 1 Gm. in 20 mls of water with 5 mls nitric acid should show no red colour on the addition of 1 mil potassium thiocyanate solution.

**SODIUM NITRITE.****Tests.**

*Solubility:* Soluble in less than 2 parts of water.

*Sulphate:* 1 Gm. in 20 mls of water and 1 mil nitric acid should give no turbidity on the addition of 1 mil barium chloride solution.

*Chlorides:* 1 Gm. in 20 mls of water with 1 mil nitric acid should give no precipitate on the addition of 1 mil silver nitrate solution.

*Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution or 1 drop ammonium sulphide solution.

*Quantitative Determination:* Titration with N/10 permanganate solution should indicate not less than 98 per cent. sodium nitrite.

**Trade Varieties.**

Sodium nitrite is now prepared of a fair degree of purity on a very large scale for technical purposes. It is usually not quite colourless, and may contain as much as 95 per cent.  $\text{NaNO}_2$ , the chief impurities being nitrate, with traces of sulphate and chloride.

**SODIUM PHOSPHATE.**

Colourless, transparent, efflorescent crystals. The aqueous solution has an alkaline reaction.

**Tests.**

*Solubility:* Soluble in 6 parts of water, insoluble in alcohol.

*Chlorides:* 1 Gm. in 20 mls of water with 1 mil nitric acid should give no turbidity or only the faintest opalescence on the addition of 1 mil silver nitrate solution.

*Sulphates:* 1 Gm. in 20 mls of water with 1 mil hydrochloric acid should give no turbidity on the addition of 1 mil barium chloride solution and setting aside one hour.

*Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution or 1 drop ammonium sulphide solution.

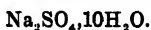
*Arsenic:* 1 Gm. introduced into the Marsh-Berzelius apparatus should give no arsenical stain after one hour's treatment.

**Trade Varieties.**

The sodium phosphate sold for medicinal purposes is of very fair purity, and contains, as a rule, only traces of sulphates and chlorides.





**SODIUM SULPHATE.**

Colourless transparent crystals, readily efflorescent.

**Tests.**

*Solubility*: Soluble in about 3 parts of water, insoluble in alcohol.

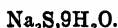
*Chlorides*: 1 Gm. in 20 mls of water should show no turbidity on the addition of 1 mil silver nitrate solution.

*Heavy Metals*: 1 Gm. in 20 mls of water should show no darkening on the addition of an equivalent volume of hydrogen sulphide solution.

*Iron*: 1 Gm. in 20 mls of water with three drops nitric acid should not show a pink tint on the addition of 1 mil potassium sulphocyanide solution.

**Trade Varieties.**

Sodium sulphate in the form of glauber salt finds very extensive use in medicine. The medicinal variety is usually fairly pure. The salt can also be purchased in light feathery crystals, also in acicular crystals resembling magnesium sulphate and known as "Mock Epsoms."

**SODIUM SULPHIDE.**

Large, colourless, transparent crystals, which are deliquescent and become discoloured on exposure to the air.

**Tests.**

*Solubility*: 5 Gms. should dissolve in 10 mls of water, forming a clear solution.

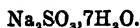
*Other Sulphur Compounds*: 1 Gm. in 20 mls of water treated with 2 mls hydrochloric acid should evolve hydrogen sulphide and form a solution which is only faintly opalescent.

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**Trade Varieties.**

Crude sodium sulphide for technical purposes is sold in brownish crystalline masses, which are usually damp from partial deliquescence. The recrystallised variety is also available, which is not quite colourless and less pure than the variety described above. The salt is sometimes described as sodium monosulphide to distinguish it from sulphurated soda or liver of sulphur, containing poly-sulphides. The anhydrous salt is also sold, but the compound suffers much damage in the process of drying.

•

**SODIUM SULPHITE.\***

Colourless transparent prismatic crystals. Exposed to the air the salt effloresces and is slightly oxidised to sulphate.

**Tests.**

*Solubility*: Soluble in 3 parts of water, insoluble in alcohol.

*Quantitative Determination*: Dissolved in excess of N/10 iodine solution and titrated back with N/10 sodium thiosulphate solution the salt should indicate not less than 97 per cent.  $\text{Na}_2\text{SO}_3, 7\text{H}_2\text{O}$ .

**Trade Varieties.**

Commercial sodium sulphite rarely reaches the standard described above, and may fall much lower owing to oxidation if not carefully stored. The anhydrous sulphite is also sold, but suffers considerable change to sulphate during the process of drying.

**SODIUM THIOSULPHATE.**

Clear glassy crystals, when heated the salt fuses to a clear liquid.

**Tests.**

*Solubility*: Soluble in less than its own weight of water.

*Sulphide*: 1 Gm. in 20 mls of water should give a white precipitate free from any brown or black tint on the addition of five drops silver nitrate solution.

*Calcium*: 1 Gm. in 20 mls of water should give no immediate turbidity on the addition of 1 mil ammonium oxalate solution.

*Sulphate and Sulphite*: Dissolve 1 Gm. in 5 mls of water, and add decinormal iodine solution until the thiosulphate is entirely converted into tetrathionate and excess of iodine remains, this solution should give no immediate turbidity on the addition of 2 mls barium chloride solution.

**Trade Varieties.**

Commercial thiosulphate of soda is met with in many varieties, but the pea crystals are the most convenient form for general use. It is usually of very satisfactory purity.





■

**TIN.**

White soft metal melting at  $235^{\circ}$ . It is dissolved by hot hydrochloric acid, but converted into white, insoluble stannic oxide by the addition of nitric acid.

**Tests.**

*Foreign Metals:* Place 5 Gms. of the metal in a test-tube and add strong nitric acid in the proportion of 10 mils, heating gently until the whole of the metal has been attacked; evaporate on a water-bath to dryness and triturate the residue with 50 mils of water and 2 mils nitric acid; filter, and divide the filtrate into four portions. Evaporate one to dryness with the addition of 5 drops sulphuric acid; the residue should be entirely soluble in water. To another portion add ammonia to distinctly alkaline reaction; the liquid should be free from blue colour. To a third portion add ammonia to alkaline reaction, and then 5 drops ammonium sulphide solution; the liquids should show at most a black opalescence indicating the presence of only traces of iron.

**Trade Varieties.**

Tin for laboratory use is sold granulated and in cylindrical rods. Commercial tin known as grain tin is in the form of long thin sticks, which are easily bent and emit a peculiar sound known as the "cry" of tin. The cruder varieties are very impure.

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**TIN CHLORIDE.**

Stannous Chloride,  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ .

White, semi-opaque crystals, which dissolve to a clear solution in water containing hydrochloric acid, but are decomposed by pure water with formation of basic salt.

**Tests.**

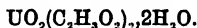
*Foreign Metals:* Dissolve 1 Gm. in a mixture of 10 mils water and 3 mils hydrochloric acid; dilute the product to 100 mils, and completely precipitate the tin with hydrogen sulphide. The filtrate evaporated to dryness should leave no residue or one weighing less than 1 Mg.

*Sulphate:* Dissolve 1 Gm. in a mixture of 10 mils water and 3 mils hydrochloric acid; dilute to 50 mils with distilled water, and add 1 mil barium chloride solution; no immediate turbidity should be produced.

*Arsenic:* Place 3 Gms. with 20 mils hydrochloric acid in a wide test-tube and immerse the tube in a beaker of boiling water; the solution should remain clear and colourless after half an hour's immersion.

**Trade Varieties**

Commercial stannous chloride, known as "tin crystals," is used largely for technical purposes. The crystals are usually more or less discoloured, and do not form a perfectly clear solution in dilute hydrochloric acid.

**URANIUM ACETATE.**

Small yellow crystals having an acetous odour.

**Tests.**

*Solubility:* 1 Gm. dissolved in 25 mls of water forms a clear solution or one having a faint opalescence, which is removed with the addition of a few drops of acetic acid.

*Sulphates:* 0.5 Gm. dissolved in 20 mls of water should give no immediate turbidity on the addition of 1 ml barium chloride solution.

*Alkalies:* 1 Gm. dissolved in 25 mls of water heated to boiling and precipitated by the addition of excess of ammonia solution. The filtrate on evaporation and ignition should yield a residue weighing not more than 4 Mgs.

*Calcium:* 1 Gm. in 25 mls of water should give a clear solution on the addition of excess of ammonium carbonate solution; about 15 mls should be required.

*Metals:* The liquid from the preceding test should show no darkening on the addition of 3 drops ammonium sulphide solution previously distilled with 5 mls of water.

**Trade Varieties.**

Ordinary uranium acetate of trade usually contains considerable quantities of sodium acetate.

**URANIUM NITRATE.**

Yellowish-green translucent crystals.

**Tests.**

*Solubility:* 1 Gm. with 20 mls of water form a clear solution having an acid reaction.

*Impurities:* These can be tested for in the manner described under "Uranium Acetate."

**Trade Varieties.**

The usual impurities to be found are sodium nitrate, traces of iron, calcium, and sulphate.





**ZINC.**

Bluish-white metal, which is supplied in the form of granules, coarse powder, or rods. For analytical purposes the two following grades are usually employed :—

(a) *Zinc for forensic purposes*: This should contain almost inappreciable traces of iron and less than 1 in 2,000,000 of arsenic when tested as described below. Further, 10 Gms. when completely dissolved in dilute sulphuric acid should leave only a few minute black specks of insoluble matter.

**Tests.**

*Arsenic*: Add 15 mls sulphuric acid cautiously to 60 mls of water. Cool and place with 12 Gms. of the zinc in a Marsh-Berzelius apparatus. Continue the action for forty-five minutes, or until the zinc has almost completely dissolved; the arsenical stain, if any, should be less than that corresponding to 1 part in 2,000,000 of arsenic, as determined by comparison with a standard arsenical tube. Many analysts prefer to add 5 mls of a 33 per cent. solution of cadmium sulphate, which is claimed to render the detection of the arsenic more certain, and to facilitate the action of the acid.

*Iron*: The contents of the Marsh-Berzelius bottle should be carefully decanted from any insoluble matter. The addition of 0.1 ml N/10 potass. permanganate solution should render the liquid distinctly pink.

(b) *Zinc Chemically Pure*: This variety should correspond with the tests as described above except that the limit of arsenic may be reduced to 1 in 1,000,000.

**Trade Varieties.**

Commercial zinc as used for industrial purposes contains considerable traces of other metals, chiefly iron and lead with some carbon. The two last-mentioned will be found in the insoluble residue left after dissolving the zinc in acid, and the excessive quantities of iron will be revealed by the large quantity of permanganate necessary to give the liquid a pink tint. The ordinary zinc sold as "free from arsenic" sometimes contains considerable quantities of iron which render the zinc insensitive for arsenic determinations.

**ZINC CHLORIDE.**

A white deliquescent salt, usually sold in the form of cakes or sticks.

**Tests.**

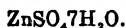
*Solubility:* The salt dissolves in its own weight of water, forming a clear solution, having an acid reaction, which is rendered opalescent on further dilution. The opalescence is removed by the addition of hydrochloric acid.

*Sulphates:* Dissolve 1 Gm. in 10 mls of water and just sufficient hydrochloric acid to make a clear solution; no immediate turbidity should appear on the addition of 1 mil barium nitrate solution.

*Heavy Metals:* 1 Gm. in 20 mls of water with sufficient hydrochloric acid to give a clear solution should show no darkening on the addition of an equal volume hydrogen sulphide solution.

**Trade Varieties.**

Zinc chloride in the form of nearly white or greyish-white lumps or powder has many technical applications, and contains usually traces of other metals and considerable quantities of iron as well as sulphate.

**ZINC SULPHATE.**

Colourless needle crystals, efflorescent, insoluble in alcohol.

**Tests.**

*Solubility:* Zinc sulphate is soluble in less than its own weight of distilled water, and should form a clear solution with 10 parts of water having an acid reaction.

*Chlorides:* 1 Gm. dissolved in 20 mls of water should give no immediate turbidity on the addition of 1 mil silver nitrate solution.

*Heavy Metals:* 1 Gm. in 20 mls of water should show no darkening on the addition of an equal volume of hydrogen sulphide solution.

*Iron:* 0.5 Gm. in 30 mls of water with 1 mil hydrochloric acid should not be reddened by the addition of 1 mil potassium thiocyanate solution.

*Arsenic:* 1 Gm. should give no arsenical mirror after thirty minutes' action in the Marsh-Berzelius apparatus.

**Trade Varieties.**

Commercial zinc sulphate is often of very fair purity if white and well crystallised. Cruder varieties are often greyish and moist, with an acid mother liquor. The chief impurities to be expected are arsenic and iron.







## Appendix :

### Preparation of Solutions to be Used in the Tests.

In order to give the tests and reactions, described in the series of articles which have appeared under the above title, a definite limiting value it is necessary to define the concentration and composition of the various test solutions which have been mentioned. It has been customary hitherto to employ solutions of various strengths, as is done in the Appendix to the British Pharmacopœia, and to select a concentration guided by the solubility of the various salts employed and by the nature of the reactions in which their solutions were to be employed. The idea of using normal solutions in qualitative tests has recently been revived.\* Their use undoubtedly serves to give a certain precision to qualitative reactions which is not afforded by solutions having an arbitrarily selected concentration—e.g., 5, 10, or 20 per cent. The advantage is most marked in the case of the dilute acids and the solutions of the alkaline hydroxides. If, for example, we prepare these solutions of the same normality, then equal volumes of such solutions will have an equal value in chemical reactions, although the actual percentage of each chemical substance will be different. Thus if we have employed a known volume of 2N/1 nitric acid to render a solution acid we know then an equal volume of 2N/1 sodium hydroxide will bring the liquid back to neutrality. It is not necessary for these purposes to standardise the solutions; it suffices to weigh carefully the necessary quantity of substance known to be pure and dissolve it in the solvent to the required volume. Again, if a given quantity of N/1 barium chloride solution has been employed it can be seen at once that a slightly larger quantity of N/1 sodium sulphate solution will provide, under suitable conditions, for the removal from the solution of all the barium as sulphate. In the test solutions described below the selection of the normality follows the conventional and arbitrary standards hitherto employed as closely as is consistent with the principles laid down.

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\* *Exronlisse*, 'Proceedings Brussels International Congress of Pharmacy 1910.'

**A.—Concentrated Acids.**

Acetic acid, N17.5/1, specific gravity 1.058, containing 99 per cent.  $\text{HC}_2\text{H}_3\text{O}_2$ .

Hydrochloric acid, N10.1/1, specific gravity 1.16, containing 31.79 per cent.  $\text{HCl}$ .

Nitric acid, N15.8/1, specific gravity 1.42, containing 70 per cent.  $\text{HNO}_3$ .

Sulphuric acid, N36.86/1, specific gravity 1.843, containing 98 per cent.  $\text{H}_2\text{SO}_4$ .

**B.—Dilute Acids, 2N/1 Normal.**

Dilute acetic acid, 2N/1. 1 litre contains 120 Gms.  $\text{HC}_2\text{H}_3\text{O}_2$ . Dilute 121.8 Gms. concentrated acetic acid to 1 litre.

Dilute hydrochloric acid, 2N/1. 1 litre contains 73 Gms.  $\text{HCl}$ . Dilute 229.6 Gms. (198 mls) concentrated acid to 1 litre.

Dilute nitric acid, 2N/1. 1 litre contains 126 Gms.  $\text{HNO}_3$ . Dilute 180 Gms. (127 mls) concentrated acid to 1 litre.

Dilute sulphuric acid, 2N/1. 1 litre contains 98 Gms.  $\text{H}_2\text{SO}_4$ . Dilute 100 Gms. (54.4 mls) concentrated acid to 1 litre.

Tartaric acid solution, 2N/1. 1 litre contains 150 Gms.  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ .

**C.—Concentrated Alkali.**

Stronger solution of ammonia, N16.5/1, specific gravity 0.891, containing 31.5 per cent.  $\text{NH}_3$ .

**D.—Alkaline Solutions, 2N/1 Normal.**

Dilute ammonia solution, 2N/1. 1 litre contains 34 Gms.  $\text{NH}_3$ . Dilute 107.9 Gms. (121 mls) of stronger solution of ammonia to 1 litre.

Sodium hydroxide solution, 2N/1. 1 litre contains 80 Gms.  $\text{NaOH}$ . Dissolve 85 Gms. pure sodium hydroxide (containing about 95 per cent.  $\text{NaOH}$ ) to 1 litre.

Potassium hydroxide solution, 2N/1. 1 litre contains 112 Gms.  $\text{KOH}$ . Dissolve 132 Gms. pure potassium hydroxide (containing about 85 per cent.  $\text{KOH}$ ) to 1 litre.

Ammonium carbonate solution, 2N/1. 1 litre contains 96 Gms.  $(\text{NH}_4)_2\text{CO}_3$ . Dissolve 78.5 Gms. ammonium carbonate in water, add 50 mls stronger solution of ammonia, and dilute to 1 litre.

Sodium carbonate solution, 2N/1. 1 litre contains 106 Gms.  $\text{Na}_2\text{CO}_3$ , or 286 Gms.  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ . Dissolve 106 Gms. anhydrous sodium carbonate, or 286 Gms. sodium carbonate crystals, to 1 litre.





Potassium carbonate solution, 2N/1. 1 litre contains 138.3 Gms. anhydrous  $K_2CO_3$ .

## E.—Salt Solutions and Solutions of Gases and Other Substances.

Ammonium acetate solution N/1. 1 litre contains 77 Gms.  $NH_4C_2H_3O_2$ .

Ammonium chloride solution 2N/1. 1 litre contains 107 Gms.  $NH_4Cl$ .

Ammonium oxalate solution N/2. 1 litre contains 35.5 Gms.  $(NH_4)_2C_2O_4 \cdot H_2O$ .

Ammonium sulphide solution 2N. 1 litre contains 68 Gms.  $(NH_4)_2S$ . The solution of ammonium sulphide, prepared by saturating three volumes of 10 per cent. ammonia solution with hydrogen sulphide and adding two volumes of 10 per cent. ammonia solution, may be diluted by the addition of twice its volume of water.

Ammonium thiocyanate solution N/2. 1 litre contains 38 Gms.  $NH_4CNS$ .

Barium chloride solution N/2. 1 litre contains 61 Gms.  $BaCl_2 \cdot 2H_2O$ .

Barium nitrate solution N/2. 1 litre contains 65.4 Gms.  $Ba(NO_3)_2$ .

Bromine solution, about N/4. Contains 20 Gms. (6.6 mils) bromine per litre.

Cadmium iodide solution N/5. 1 litre contains 36.6 Gms.  $CdI_2$ .

Calcium chloride solution N/1. 1 litre contains 55.5 Gms. anhydrous  $CaCl_2$ , or 109.5 Gms. crystallised  $CaCl_2 \cdot 6H_2O$ .

Calcium hydroxide solution, about N/35. A saturated solution of calcium hydroxide in water contains about 1.1 Gms.  $Ca(OH)_2$  per litre.

Calcium sulphate solution, about N/34. A saturated solution of calcium sulphate in water contains about 2 Gms.  $CaSO_4$  per litre.

Chlorine solution, about N/7. Contains about 5 Gms.  $Cl$  per litre.

Copper sulphate solution N/2. 1 litre contains 62.5 Gms.  $CuSO_4 \cdot 5H_2O$ .

Ferric chloride solution N/1. 1 litre contains 54 Gms. anhydrous  $FeCl_3$ , or 90 Gms.  $FeCl_3 \cdot 6H_2O$ .

Hydrogen sulphide solution, about N/4. 1 litre contains about 4.5 Gms.  $H_2S$ .

Lead acetate solution, N/2. 1 litre contains 94.8 Gms.  $Pb(C_2H_3O_2)_2 \cdot 3H_2O$ .

Magnesium sulphate solution N/1. 1 litre contains 123 Gms.  $MgSO_4 \cdot 7H_2O$ .

Mercuric chloride solution N/4. 1 litre contains 33.7 Gms.  $\text{HgCl}_2$ .

Mercurous nitrate solution N/10. 1 litre contains 28 Gms.  $\text{HgNO}_3, \text{H}_2\text{O}$ .

Platinic chloride solution. 1 litre contains 133 Gms.  $\text{H}_2\text{PtCl}_6, 6\text{H}_2\text{O}$ .

Potassium acetate solution N/1. 1 litre contains 98 Gms.  $\text{KC}_2\text{H}_3\text{O}_2$ . This quantity corresponds closely to 100 Gms. of freshly fused potassium acetate.

Potassium chromate N/1. 1 litre contains 97.2 Gms.  $\text{K}_2\text{CrO}_4$ .

Potassium cyanide solution N/1. 1 litre contains 65 Gms.  $\text{KCN}$ . About 67 Gms. crystallised potassium cyanide should be used.

Potassium ferricyanide solution. 1 litre contains 50 Gms.  $\text{K}_3\text{Fe}_2(\text{CN})_{12}$ .

Potassium ferrocyanide solution. 1 litre contains 50 Gms.  $\text{K}_4\text{Fe}(\text{CN})_6, 3\text{H}_2\text{O}$ .

Potassium iodide solution N/2. 1 litre contains 83 Gms.  $\text{KI}$ .

Potassium permanganate solution N/10. 1 litre contains 3.16 Gms.  $\text{KMnO}_4$ .

Silver nitrate solution N/5. 1 litre contains 34 Gms.  $\text{AgNO}_3$ .

Sodium acetate solution N/1. 1 litre contains 136 Gms.  $\text{NaC}_2\text{H}_3\text{O}_2, 3\text{H}_2\text{O}$ .

Sodium phosphate solution N/1. 1 litre contains 119.5 Gms.,  $\text{Na}_2\text{HPO}_4, 12\text{H}_2\text{O}$ .

Sodium sulphate solution N/1. 1 litre contains 161 Gms.  $\text{Na}_2\text{SO}_4, 10\text{H}_2\text{O}$ .

Stannous chloride solution N/1. 1 litre contains 113 Gms.  $\text{SnCl}_2, 2\text{H}_2\text{O}$ .

Sulphurous acid solution, about 3N/2. Saturated solution of sulphur dioxide in water. Contains about 64 Gms.  $\text{H}_2\text{SO}_3$ , or 50 Gms.  $\text{SO}_2$  per litre.







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